Results of Proficiency Test
Nickel release and
Surface determination
June 2021

Organized 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 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 jewelry in piercings (earrings), other jewelry, watches or clothes fasteners, such as buttons and belts.

Since 2014 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Nickel release and Surface determination every year. During the annual proficiency testing program 2020/2021 it was decided to continue the proficiency test for the analysis of Nickel release and Surface determination.

In this interlaboratory study 99 laboratories in 24 different countries registered for participation. See appendix 5 for the number of participants per country. In this report the test results of this proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send three identical non-coated metal plates labelled #21610 positive on Nickel release and one piece of a metal necklace chain labelled #21611 for Surface determination only. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization 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 June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

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

Nickel release Determination

A batch of Nickel containing metal was purchased from a local supplier and consisted of square metal pieces with a hole in one of the corners. The pieces were solid metal, prepared from one alloy and non-coated. The dimensions of each item were approximately 2 x 2 x 0.2 cm and the hole had a diameter of approximately 0.5 cm. Three items were packed in separate bags to avoid scratching of the items and all three together packed in a small plastic bag. The batch was divided over 150 subsamples and each bag was labelled #21610.

The homogeneity of the subsamples was checked by determination of Nickel release using test method EN1811:11+A1:15 on eight stratified randomly selected subsamples. Please note, a subsample is one bag with three items.

	Nickel release in µg/cm^2/week
sample #21610-1	1.08
sample #21610-2	1.19
sample #21610-3	1.04
sample #21610-4	1.27
sample #21610-5	1.14
sample #21610-6	1.06
sample #21610-7	1.09
sample #21610-8	1.05

Table 1: homogeneity test results of subsamples #21610

From the above test results the relative standard deviation was calculated and compared with 0.3 times the relative standard deviation, estimated from the average of PT uncertainties of previous PTs in agreement with the procedure of ISO13528, Annex B2, in the next table.

	Nickel release
RSD (observed)	7.1%
reference method	iis PTs
0.3 x RSD (reference method)	9.7%

Table 2: evaluation of the repeatability of subsamples #21610

The calculated repeatability was in agreement with 0.3 times the target relative standard deviation. Therefore, homogeneity of the subsamples was assumed.

Surface determination
A batch of metal necklace chains were obtained from a local supplier. From this batch 151 small plastic bags were filled with one piece of necklace chain consisting of approximately 20 links each and labelled #21611. No homogeneity tests were done over the subsamples because only surface determination has been requested for this sample. However, each sample was weighed in advance to ensure no large differences between subsamples.

To each of the participating laboratories one sample #21610 and one sample #21611 were sent on May 19, 2021.

2.5 ANALYZES

The participants were requested to determine Nickel release on sample #21610 and to determine surface only on sample #21611, applying the analysis procedure that is routinely used in the laboratory. It was also requested to report if the laboratory was accredited for the determination of Nickel release and to report some analytical details.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendix 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test 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 reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline

were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization 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 June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test 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. If a dataset does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize 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 test results are plotted. The corresponding laboratory numbers are on 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 reference test method. 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. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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 should be done in order to evaluate whether the reported test results are fit-for-purpose.

The z-scores were calculated according to:

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples. Five participants reported test results after the final reporting date and three other participants did not report any test results at all. In total 96 participants reported 191 test results for Nickel release and Surface determination. Observed were 7 outlying test results, which is 3.7%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all 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.

4.1 EVALUATION PER SAMPLE

In this section the reported test results are discussed per sample. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the reported test results. The abbreviations, used in these tables, are explained in appendix 6.

Test method EN1811:11+A1:15 does not have a true precision statement that mentions a repeatability and/or a reproducibility. In Annex A is mentioned that the measurement uncertainty in a 2008 interlaboratory study was 46%, while in Annex B is stated “The relative test method reproducibility in this ILC was 33.3%”. Both variations could not be met by far in previous iis PTS. Therefore, it was decided to use a target reproducibility derived from the Horwitz equation. This target is dependent on the measured Nickel concentration, surface area and ranges from 54% at 0.3 µg Ni/cm^2/week up to 32% at 10 µg Ni/cm^2/week.

Sample #21610

Nickel release: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated from the Horwitz equation.

Sample #21611

Surface determination: This determination on the necklace chain may be problematic. Six statistical outliers were observed in the reported range of 11.7-162.17 mm^2. No official test method exists for Surface determination. Therefore, no z-scores were calculated. The variation for this sample of 7% is within the range of the observed variation in previous PT’s in which the Surface determination was evaluated (3.5-13%) but is larger compared to the variation of the Surface determination of the much simpler shaped sample #21610 (0.7%).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility estimated using the Horwitz equation are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(target)
Nickel release	µg/cm^2/week	94	0.91	0.53	0.41
Surface determination	cm^2	91	9.55	0.19	n.a.

Table 3: reproducibilities of test results on sample #21610

It can be concluded, without further statistical calculations, that the group of participating laboratories had problems with the analysis of Nickel release.

Parameter	unit	n	average	2.8 * sd	R(target)
Surface determination	mm^2	90	110.2	21.3	n.a.

Table 4: reproducibility of test results on sample #21611

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2021 WITH PREVIOUS PTS

	June 2021	June 2020	June 2019 *)	May 2018 *)	May 2017 *)
Number of reporting laboratories	96	104	127	113	122
Number of test results	191	205	126	112	122
Number of statistical outliers	7	11	5	4	14
Percentage statistical outliers	3.7%	5.4%	4.0%	3.6%	11%

Table 5: comparison with previous proficiency tests

*) Nickel release determination only

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

The performance of the determination of the proficiency test was compared, expressed as relative standard deviations, see next table.

	June 2021	June 2020	June 2019	May 2018	2017-2014
Nickel release	21%	29%	30%	44%	18 - 31%
Surface determination	0.7 – 6.9%	2.5 – 3.5%	1.1 - 7%	1.3 - 13%	1.3 - 10%

Table 6: comparison of uncertainties of current PT with previous PTs

The uncertainty of the Nickel release determination did improve in comparison with the average uncertainty from the previous years.

When the results of the uncertainty of the Surface determination of the 2021 PT is compared with the 2020 PT, it can be concluded that (0.7%) for sample #21610 this is lower than before (0.7% vs 2.5% in the PT of 2020), while for sample #21611 it was higher than before (6.9% vs 3.5% in the PT of 2020). The latter probably due to the more complex shape of the sample than in 2020.

4.4 Evaluation of the Analytical Details

For the Nickel release sample #21610 the following can be summarized from the various analytical details provided by the participants (these are given in appendix 2 and 3):

- About 90% of the reporting laboratories are accredited for the determination of Nickel release.
- About 95% of the reporting participants used all three plates for the Nickel release determination.
- About 55% of the reporting participants have used new or disposable test vessels, while 35% of the reporting participants done a pre-treatment of the test vessels. Remarkably, 6% of the reporting participants have not done any pre-treatment.
- About 80% of the reporting participants used a ratio of approximately 1mL test solution per cm^2 sample surface area. Remarkedly, a few participants reported to have used a volume to surface ratio of 1.5 to 4 mL per cm^2.

For the Surface determination sample #21611 only one question was requested: a detailed description on how the surface area was measured and calculated which was answered by approximately 65% of the reporting participants. A variety of methods was given, see appendix 4 for the answers given.

5 Discussion

The variation on the Nickel release results for the individual items was large; with an RSD from 0.2% up to 38.0%. Only 37 participants had an RSDr in agreement with the target repeatability standard deviation of 4%, estimated from EN1811:11+A1:15 (33.3% / 2.8 / 3). Remarkedly, when evaluating the test results of only those 37 participants the average and variation are not significant different from the total group. See appendix 1 for this analysis.

Some participants used new or disposable test vessels for which pre-treatment is not necessary. Test vessels that have been used before should be pre-treated with 5% Nitric acid for at least 4 hours, see paragraph 6.4 of EN1811:11+A1:15. This is necessary to remove any Nickel present from earlier use. When used vessels are not pre-treated, there will be a risk that the test result for Nickel release will be higher. However, the investigated effect of pre-treatment vs. non pre-treatment was very low and not significant in this year's proficiency test.

It was observed that a number of participants possibly reported the end volume after dilution, e.g. 25mL. Test method EN1811:11+A1:15 prescribes the amount of test solution to be used to be 1mL per cm^2 surface area, which in this PT is between 9 and 10 mL. Not all participants used this ratio. However, when evaluating the test results of only those participants that used an 1:1 ratio for test solution to surface area than the average and variation are not significant different from the total group. See appendix 1 for this analysis.

6 CONCLUSION

It can be concluded that the majority of the participants do not have a problem with the determination on Nickel release, each participating laboratory needs to evaluate its performance in this study and decide about any corrective actions if necessary.

Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and increase the quality of the analytical results.

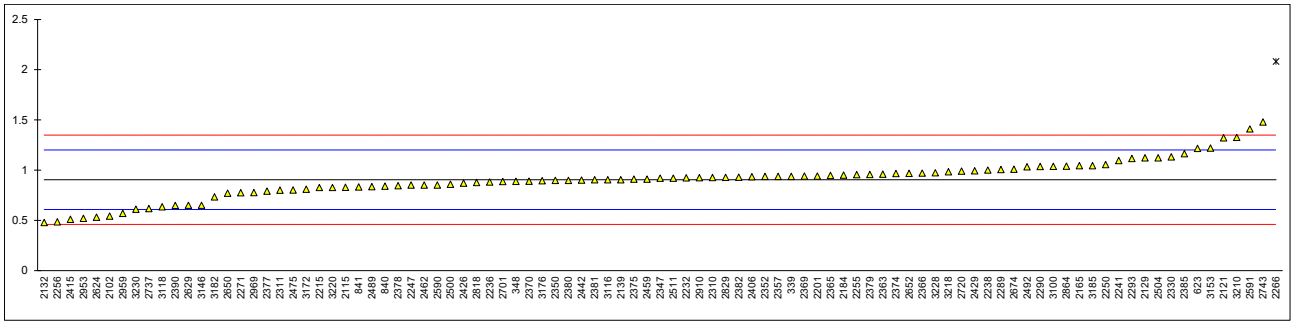
APPENDIX 1

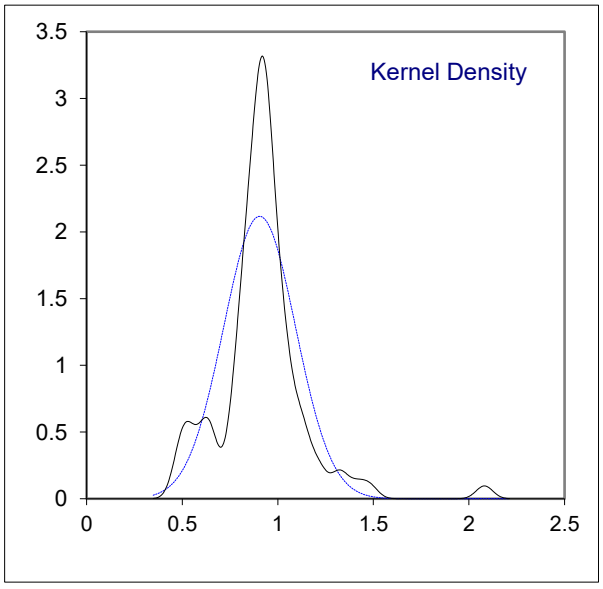
Determination of Nickel release on sample #21610; average result of three replicates in µg/cm²/week

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210		-----		-----	
339	EN1811 + A1	0.93702		0.21	
348	EN1811 + A1	0.888		-0.12	
623	EN1811 + A1	1.216		2.10	
840	EN1811 + A1	0.84		-0.44	
841	EN1811 + A1	0.832		-0.49	
2102	EN1811	0.543		-2.45	
2115	EN1811	0.83		-0.51	
2121	EN1811 + A1	1.32	C	2.80	first reported: 1.49
2129	EN1811 + A1	1.122		1.46	
2132	EN1811 + A1	0.4794		-2.88	
2139	EN1811	0.904	C	-0.01	first reported: 1.402
2165	EN1811 + A1	1.044		0.94	
2184	EN1811 + A1	0.950		0.30	
2201	EN1811 + A1	0.9402		0.24	
2215	EN1811	0.8267	C	-0.53	first reported: 0.8561
2232	EN1811 + A1	0.923		0.12	
2236	In house	0.881		-0.16	
2238	EN1811 + A1	1.000		0.64	
2241	EN1811 + A1	1.095	C	1.28	first reported: 1.554
2247	EN1811	0.85		-0.37	
2250	EN1811	1.0553		1.01	
2255	EN1811 + A1	0.955		0.34	
2256	EN1811 + A1	0.4846		-2.84	
2266	EN1811 + A1	2.081	R(0.01)	7.94	
2271	EN1811	0.775		-0.88	
2289	EN1811 + A1	1.006		0.68	
2290	EN1811 + A1	1.036		0.88	
2293	EN1811 + A1	1.115		1.42	
2310	EN1811 + AC	0.926		0.14	
2311	EN1811 + A1	0.8012		-0.70	
2330	EN1811	1.1319		1.53	
2347	EN1811 + A1	0.92		0.10	
2350	EN1811 + A1	0.897		-0.06	
2352	EN1811	0.937		0.21	
2357	EN1811	0.937		0.21	
2363	EN1811 + A1	0.96		0.37	
2365	EN1811	0.9471		0.28	
2366	EN1811	0.97		0.44	
2369	EN1811	0.940		0.23	
2370	EN1811	0.890		-0.10	
2374	EN1811	0.966		0.41	
2375	EN1811	0.91		0.03	
2377	EN1811	0.79		-0.78	
2378	EN1811 + A1	0.845		-0.41	
2379	EN1811 + A1	0.957		0.35	
2380	EN1811 + A1	0.897		-0.06	
2381	EN1811	0.903		-0.02	
2382	EN1811 + A1	0.929		0.16	
2385	EN1811	1.164		1.75	
2390	EN1811 + A1	0.648		-1.74	
2406	EN1811 + A1	0.933		0.19	
2415	EN1811 + A1	0.510		-2.67	
2426	EN1811	0.87		-0.24	
2429	EN1811 + A1	0.992		0.59	
2442	EN1811 + A1	0.901		-0.03	
2459	EN1811	0.910		0.03	
2462	EN1811	0.850		-0.37	
2475	EN1811 + A1	0.802		-0.70	
2489	EN1811 + A1	0.8354		-0.47	
2492	In house	1.034		0.87	
2500	EN1811 + A1	0.8594		-0.31	
2504	EN1811 + A1	1.122	C	1.46	first reported: 2.013
2511	EN1811	0.92		0.10	
2582		-----		-----	
2590	EN1811	0.8507	C	-0.37	first reported: 0.3088
2591	EN1811	1.40969		3.41	
2624	EN1811	0.53		-2.53	
2629	EN1811 + A1	0.65	C	-1.72	first reported: 1.65
2650	EN1811 + A1	0.77		-0.91	
2652	EN1811	0.9663		0.41	
2674	EN1811 + A1	1.009		0.70	
2701	EN1811	0.8867		-0.13	

lab	method	value	mark	z(targ)	remarks
2720	EN1811 + A1	0.990		0.57	
2737	EN1811	0.6170		-1.95	
2743	EN1811 + A1	1.47925942		3.88	
2818	EN1811 + A1	0.876		-0.20	
2829	EN1811	0.927		0.15	
2864	EN1811 + A1	1.04		0.91	
2910	EN1811 + A1	0.924		0.13	
2953	EN1811 + A1	0.52		-2.60	
2959	EN1811	0.57		-2.26	
2969	EN1811	0.7765		-0.87	
3100	EN1811 + A1	1.0376		0.89	
3110		-----		-----	
3116	EN1811 + A1	0.9031		-0.01	
3118	EN1811 + A1	0.6346		-1.83	
3146	EN1811 + A1	0.650		-1.72	
3153	EN1811 + A1	1.220		2.13	
3172	EN1811 + A1	0.81		-0.64	
3176	EN1811	0.895		-0.07	
3182	EN1811	0.733		-1.16	
3185	EN1811 + A1	1.044		0.94	
3210	EN1811	1.325		2.84	
3218	EN1811 + A1	0.983		0.53	
3220	EN1811	0.827		-0.53	
3228	EN1811 + A1	0.973		0.46	
3230	EN1811	0.610534		-1.99	

				Only RSD <4% between		Only with ratio 1:1	
	normality	suspect		not OK		OK	
	n	94		37		66	
	outliers	1		0		1	
	mean (n)	0.905		0.929		0.904	
	st.dev. (n)	0.1884	RSD = 21%	0.1712	RSD = 18%	0.1600	RSD = 18%
	R(calc.)	0.528		0.479		0.448	
	st.dev.(Horwitz)	0.1481		0.1514		0.1479	
	R(Horwitz)	0.415		0.424		0.414	
comp							
	R(EN1811:11+A1:15)	0.301					



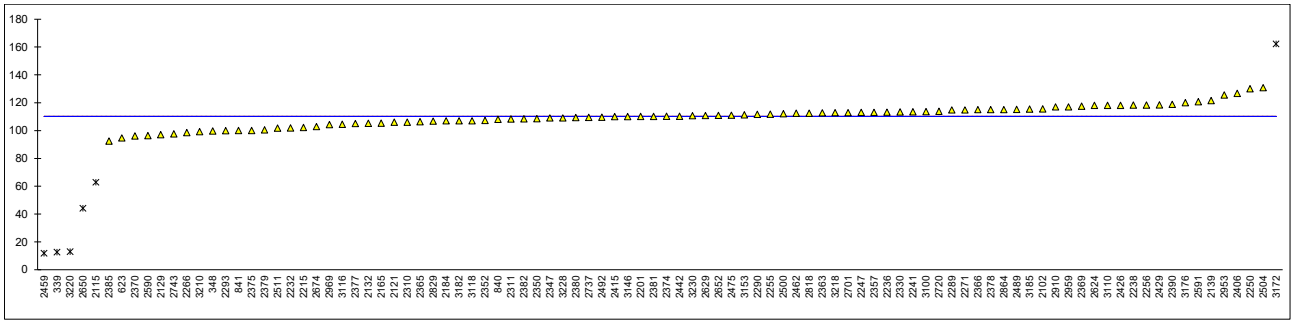


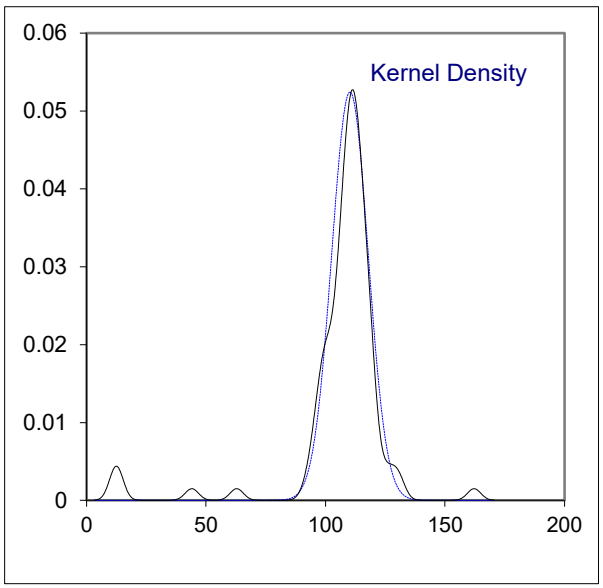
Determination of Surface on sample #21611; results in mm^2

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210		-----		-----	
339		12.6	R(0.01)	-----	
348	see appendix 4	99.67		-----	
623	see appendix 4	94.67		-----	
840		108		-----	
841	see appendix 4	100		-----	
2102	see appendix 4	115.4928		-----	
2115		62.8	R(0.01)	-----	
2121	see appendix 4	106	C	-----	first reported: 177.8
2129		97		-----	
2132	see appendix 4	105.24		-----	
2139	see appendix 4	121.518		-----	
2165	see appendix 4	105.31		-----	
2184		106.9		-----	
2201	see appendix 4	110.1198		-----	
2215	see appendix 4	102.2		-----	
2232	see appendix 4	101.806		-----	
2236	see appendix 4	113.0973		-----	
2238	see appendix 4	118.2		-----	
2241	see appendix 4	113.506		-----	
2247	see appendix 4	113.00		-----	
2250	see appendix 4	130		-----	
2255	see appendix 4	111.7		-----	
2256	see appendix 4	118.3		-----	
2266	see appendix 4	98.4		-----	
2271		114.8	C	-----	first reported: 1.148
2289	see appendix 4	114.77		-----	
2290		111.6		-----	
2293	see appendix 4	99.89		-----	
2310	see appendix 4	106		-----	
2311	see appendix 4	108.27		-----	
2330	see appendix 4	113.32		-----	
2347		109		-----	
2350	see appendix 4	108.44		-----	
2352		107.221		-----	
2357		113		-----	
2363		112.74		-----	
2365	see appendix 4	106.3		-----	
2366		115		-----	
2369		117.5		-----	
2370	see appendix 4	96.1		-----	
2374	see appendix 4	110.252		-----	
2375	see appendix 4	100.0		-----	
2377		105	C	-----	first reported: 1.05
2378		115		-----	
2379	see appendix 4	100.40	C	-----	first reported: 1.004
2380	see appendix 4	109.372		-----	
2381	see appendix 4	110.12		-----	
2382	see appendix 4	108.3		-----	
2385	see appendix 4	92.31		-----	
2390	see appendix 4	118.774		-----	
2406	see appendix 4	126.6		-----	
2415	see appendix 4	109.96		-----	
2426	see appendix 4	118.06		-----	
2429	see appendix 4	118.32		-----	
2442	see appendix 4	110.252		-----	
2459	see appendix 4	11.7	R(0.01)	-----	
2462		112.4		-----	
2475		111		-----	
2489	see appendix 4	115.22	C	-----	first reported: 1.1522
2492	see appendix 4	109.5		-----	
2500	see appendix 4	112.1		-----	
2504	see appendix 4	130.748		-----	
2511		101.67	C	-----	first reported: 162
2582		-----		-----	
2590	see appendix 4	96.2231	C	-----	first reported: 51.3978
2591	see appendix 4	120.607		-----	
2624	see appendix 4	118	C	-----	first reported: 1.28
2629	see appendix 4	110.712	C	-----	first reported: 199.2814
2650	see appendix 4	44	C,R(0.01)	-----	first reported: 1.14
2652		110.869		-----	
2674	see appendix 4	102.841		-----	
2701		112.8257		-----	

lab	method	value	mark	z(targ)	remarks
2720	see appendix 4	113.84		-----	
2737	see appendix 4	109.4		-----	
2743		97.544		-----	
2818	see appendix 4	112.4		-----	
2829		106.7		-----	
2864	see appendix 4	115		-----	
2910	see appendix 4	116.9		-----	
2953		125.5		-----	
2959	see appendix 4	117		-----	
2969	see appendix 4	104.23		-----	
3100	see appendix 4	113.544		-----	
3110		118		-----	
3116		104.4204		-----	
3118	see appendix 4	106.955	C	-----	first reported: 213.91
3146	see appendix 4	110		-----	
3153	see appendix 4	111.3		-----	
3172	see appendix 4	162.17	R(0.01)	-----	
3176		120		-----	
3182	see appendix 4	106.936	C	-----	first reported: 224.565
3185	see appendix 4	115.4		-----	
3210		99.2		-----	
3218	see appendix 4	112.77		-----	
3220	see appendix 4	12.95	R(0.01)	-----	
3228	see appendix 4	109		-----	
3230	see appendix 4	110.598		-----	

normality	OK	
n	90	
outliers	6	
mean (n)	110.2017	
st.dev. (n)	7.60584	RSD = 6.9%
R(calc.)	21.2964	





APPENDIX 2

Determination of Nickel release and some Analytical Details on subsamples #21610

lab	value plate 1 (µg/cm2/week)		value plate 2 (µg/cm2/week)		value plate 3 (µg/cm2/week)		RSDr (%) calc. by iis	area used for calculation (cm²)		volume test solution (mL)	ratio volume vs. area (mL/cm²)
110	-----		-----		-----		-----	-----		-----	-----
210	-----		-----		-----		-----	-----		-----	-----
339	0.75305		1.1377		0.9203		16.8	9.41		9.41	1.00
348	0.883		0.831		0.951		5.5	9.528		25	2.62
623	0.88		1.36		1.41		19.6	9.626		20	2.08
840	0.88		0.89		0.75		7.6	9.53		10	1.05
841	0.780		0.885		0.832		5.2	9.56		10	1.05
2102	0.497		0.600		0.533		7.9	9.608		9.6	1.00
2115	0.93		0.77		0.80		8.3	9.49		12	1.26
2121	1.27		1.22	C	1.46	C	7.9	9.56		20	2.09
2129	1.395		0.765		1.206		23.5	9.53		11	1.15
2132	0.4569		0.4627		0.5188		5.8	9.523		9.52	1.00
2139	0.790	C	1.015	C	0.906	C	10.2	9.56749		15	1.57
2165	1.052		1.143		0.936		8.1	9.560		10	1.05
2184	0.976		0.932		0.942		2.0	9.502		10	1.05
2201	0.9272		0.9517		0.9418		1.1	9.5		9.5	1.00
2215	0.8231	C	0.8304	C	0.8267	C	0.4	9.63	C	10	1.04
2232	0.839		0.933		0.998		7.1	9.301		9.3	1.00
2236	1.009		0.824		0.810		10.3	9.554		5	0.52
2238	0.881		1.163		0.955		11.9	9.501		9.5	1.00
2241	1.217		1.036	C	1.032	C	7.9	9.556		12	1.26
2247	0.84		0.87		0.85		1.5	9.35		11	1.18
2250	1.1244		0.9580		1.0834		6.7	9.63		11	1.14
2255	0.978		1.0		0.889		5.0	9.56		10	1.05
2256	0.5082		0.5108		0.4949	C	2.4	9.5423		15	1.57
2266	1.947		2.174		2.121		4.7	9.67		9.7	1.00
2271	0.710		0.855		0.761		7.7	9.588		10	1.04
2289	0.975		1.031		1.012		2.3	9.56		9.6	1.00
2290	0.884		1.059		1.164		11.2	9.60		-----	-----
2293	0.7985		0.8325		1.7145		38.0	9.579		25	2.61
2310	0.94		0.88		0.96		3.7	9.51		9	0.95
2311	0.8029		0.7893		0.8113		1.1	9.54		9.6	1.01
2330	0.9844		1.2239		1.1875		9.3	9.5842		10	1.04
2347	0.95		0.89		0.93		2.7	9.51		9.51	1.00
2350	0.691		1.015		0.984		16.3	9.635		13	1.35
2352	0.911		0.928		0.972		2.7	9.427		9.4	1.00
2357	0.973		0.868		0.970		5.2	9.50		-----	-----
2363	1.01		1.04		0.83		9.7	9.52		9.52	1.00
2365	0.9244		0.9447		0.9723		2.1	9.620		9.62	1.00
2366	0.95		0.99		0.96		1.8	9.56		10	1.05
2369	0.945		0.934		0.940		0.5	9.560		-----	-----
2370	0.888		0.892		0.889		0.2	9.55		20	2.09
2374	0.966		0.940		0.993		2.2	9.570		9.57	1.00
2375	0.9		0.92		0.91		0.9	9.52		-----	-----
2377	0.66		0.90		0.82		12.6	9.50		-----	-----
2378	0.795		0.908		0.831		5.6	9.58		9.6	1.00
2379	0.693		1.139		1.040		20.0	9.522		10	1.05
2380	0.958		0.746		0.989		12.0	9.537		12	1.26
2381	0.960		0.850		0.899		5.0	9.516		10	1.05
2382	0.928		0.916		0.942		1.1	9.553		9.55	1.00
2385	1.235		1.331		0.926		14.8	9.56		12	1.26
2390	0.640		0.638		0.667		2.0	9.367		9.4	1.00
2406	0.789		1.135		0.876		15.7	9.643		10	1.04
2415	0.532		0.472		0.526		5.3	9.51		10	1.05
2426	0.87		0.86		0.88		0.9	9.646		-----	-----
2429	0.981		0.963		1.032		2.9	9.561		15	1.57
2442	0.914		0.909		0.881		1.6	9.5215		9.5	1.00
2459	0.880		0.921		0.930		2.4	-----		18	1.89
2462	0.901		0.816		0.832		4.3	9.507	C	10.0	1.05
2475	0.947		0.947		0.511		25.6	9.51		9.51	1.00
2489	0.8519		0.8381		0.8163		1.8	9.56	C	10	1.05
2492	0.933		1.043		1.125		7.6	9.452		12	1.27
2500	0.8612		0.8542		0.8624		0.4	9.652		10	1.04
2504	1.116	C	1.164	C	1.088	C	2.8	9.585		10	1.04
2511	0.84		0.97		0.96		6.4	9.4	C	-----	-----
2582	-----		-----		-----		-----	-----		-----	-----
2590	0.8932	C	0.9294	C	0.7295	C	10.2	9.4040		20	2.13
2591	1.33609		1.47153		1.42145		4.0	9.639		40	4.15
2624	0.62		0.39		0.59		19.1	9.53		50	5.25
2629	0.69	C	0.51	C	0.74	C	15.3	9.59		25	2.61
2650	0.82		0.74		0.75		4.6	7.6	C	4	0.53
2652	0.8798		1.0567		0.9623		7.5	9.476		-----	
2674	1.024		0.964		1.038		3.2	9.624		12	1.25
2701	0.7614		0.9093		0.9893		10.6	9.5544		-----	

lab	value plate 1 (µg/cm2/week)	value plate 2 (µg/cm2/week)	value plate 3 (µg/cm2/week)	RSDr (%) calc. by iis	area used for calculation (cm²)		volume test solution (mL)	ratio volume vs. area (mL/cm²)
2720	1.008	0.968	0.994	1.7	9.60		9.60	1.00
2737	0.4776	0.7573	0.6161	18.5	9.51		9.5	1.00
2743	1.4768005	1.4166636	1.5443141	3.5	9.49397		15.0	1.58
2818	0.825	0.886	0.918	4.4	9.541		9.54	1.00
2829	1.000	0.946	0.834	7.5	9.588		12	1.25
2864	1.08	1.09	0.94	6.6	9.53		10	1.05
2910	0.911	0.943	0.917	1.5	9.597		9.60	1.00
2953	0.47	0.52	0.58	8.6	9.72		-----	-----
2959	0.53	0.46	0.72	19.3	9.50		9.50	1.00
2969	0.9187	0.7868	0.6241	15.5	9.590		10	1.04
3100	0.9813	0.9535	1.1780	9.6	9.545		9.54	1.00
3110	0.85	0.99	0.80	-----	9.59	C	-----	-----
3116	0.9463	0.8468	0.9162	4.6	9.55		10	1.05
3118	0.6271	0.7433	0.5333	13.5	9.5547		15	1.57
3146	0.730	0.670	0.550	11.5	9.58		9.6	1.00
3153	1.160	1.125	1.376	9.1	9.563		9.6	1.00
3172	0.78	0.92	0.73	9.9	9.227		9.227	1.00
3176	0.863	0.929	0.895	3.0	9.51		12.6	1.32
3182	0.834	0.635	0.732	11.1	9.646		9.64	1.00
3185	1.047	1.015	1.070	2.2	9.578		10.0	1.04
3210	1.575	1.241	1.160	13.6	9.52		10	1.05
3218	0.963	1.002	0.984	1.6	9.60		10	1.04
3220	0.663	0.904	0.913	14.0	9.207		9	0.98
3228	0.98	0.98	0.96	1.0	9.62		9.62	1.00
3230	0.617310	0.620520	0.593773	2.0	9.34658		15	1.60

normality	suspect	
n	91	
outliers	4	
mean (n)	9.547	
st.dev. (n)	0.0673	RSD = 0.7%
R(calc.)	0.188	

Lab 2121 first reported for plate 2: 1.48 and for plate 3: 1.72
Lab 2139 first reported for plate 1: 1.248, for plate 2: 1.574 and for plate 3: 1.385
Lab 2215 first reported for plate 1: 0.8523, for plate 2: 0.8598, for plate 3: 0.8561 and for the surface area: 9.30
Lab 2141 first reported for plate 2: 1.726 and for plate 3: 1.720
Lab 2256 first reported for plate 3: 0.4949
Lab 2462 first reported for the surface area: 1.124
Lab 2489 first reported for the surface area: 9.31
Lab 2504 first reported for plate 1: 1.956, for plate 2: 2.040 and for plate 3: 2.043
Lab 2511 first reported for the surface area: 9.227
Lab 2590 first reported for plate 1: 0.3058, for plate 2: 0.3118 and for plate 3: 0.3088
Lab 2629 first reported for plate 1: 1.69, for plate 2: 1.51 and for plate 3: 1.74
Lab 2650 first reported for the surface area: 4
Lab 3110 first reported for the surface area: 1.18

APPENDIX 3

Other reported Analytical Details for sample #21610

lab	ISO17043 accredited	pre-cleaning of test vessel	cleaning solution
110	---	---	
210	---	---	
339	No	No, new/disposable test vessel(s) were used	
348	Yes	No, new/disposable test vessel(s) were used	
623	Yes	Yes, the previously used test vessel(s) were pre-treated	sink in acid bath and rinse with distillated water and store for Ni release test.
840	Yes	Yes, the previously used test vessel(s) were pre-treated	soak with the HNO3 10% about 24h
841	Yes	Yes, the previously used test vessel(s) were pre-treated	immerse the test vessel in diluted acid nitric solution at least 4 hours, wash off with deion water then drying.
2102	Yes	Yes, the previously used test vessel(s) were pre-treated	The vessels are cleaned with 1% nitric acid during one night, rinsed with water and dried.
2115	Yes	No, the previously used test vessel(s) were not pre-treated	
2121	No	Yes, the previously used test vessel(s) were pre-treated	Degreasing with sodium dodecylbenzenesulfate and deionised water
2129	Yes	No, new/disposable test vessel(s) were used	
2132	Yes	No, new/disposable test vessel(s) were used	
2139	Yes	Yes, the previously used test vessel(s) were pre-treated	The vessels were pre-treated by being stored in a solution of dilute acid for 4hrs. And rinse the vessel with deionised water and dry.
2165	Yes	No, the previously used test vessel(s) were not pre-treated	
2184	Yes	No, new/disposable test vessel(s) were used	
2201	Yes	Yes, the previously used test vessel(s) were pre-treated	Nitric acid dilute 5%,cleaning 4 hours
2215	Yes	Yes, the previously used test vessel(s) were pre-treated	Stored in 5% HNO3 for 4H.
2232	Yes	Yes, the previously used test vessel(s) were pre-treated	Pre-treated in a solution of diluted nitric acid for at least 4 hours. After acid treatment, rinse the vessel with deionized water and dry.
2236	Yes	No, new/disposable test vessel(s) were used	
2238	Yes	No, new/disposable test vessel(s) were used	
2241	Yes	No, new/disposable test vessel(s) were used	
2247	Yes	No, new/disposable test vessel(s) were used	
2250	Yes	No, new/disposable test vessel(s) were used	
2255	Yes	No, new/disposable test vessel(s) were used	
2256	Yes	No, new/disposable test vessel(s) were used	
2266	No	No, new/disposable test vessel(s) were used	
2271	Yes	Yes, the previously used test vessel(s) were pre-treated	5%(m/m) Nitric acid 12h
2289	Yes	No, new/disposable test vessel(s) were used	
2290	Yes	Yes, the previously used test vessel(s) were pre-treated	
2293	Yes	No, new/disposable test vessel(s) were used	
2310	Yes	Yes, the previously used test vessel(s) were pre-treated	Dil.nitric acid and then rinsed with deionized water and dry the vessel
2311	Yes	Yes, the previously used test vessel(s) were pre-treated	Pretreat with 5% Nitric acid, rinsed with deionized water and dried.
2330	No	Yes, the previously used test vessel(s) were pre-treated	Immerse in 10% Nitric acid over night
2347	Yes	No, new/disposable test vessel(s) were used	
2350	Yes	No, new/disposable test vessel(s) were used	
2352	Yes	No, new/disposable test vessel(s) were used	
2357	---	---	
2363	Yes	Yes, the previously used test vessel(s) were pre-treated	use 5% HNO3 stay for 4 hours
2365	Yes	No, new/disposable test vessel(s) were used	
2366	Yes	Yes, the previously used test vessel(s) were pre-treated	Wash after soaking in acid
2369	Yes	---	
2370	Yes	No, new/disposable test vessel(s) were used	
2374	Yes	No, the previously used test vessel(s) were not pre-treated	
2375	Yes	---	-
2377	---	---	
2378	Yes	No, new/disposable test vessel(s) were used	
2379	Yes	Yes, the previously used test vessel(s) were pre-treated	5% HNO3 / 4 hr
2380	Yes	Yes, the previously used test vessel(s) were pre-treated	Firstly, all glass vessels washed by detergent powder & water. Secondly, all vessels pre-treated by being stored in dilute nitric acid for one night. Then rinse with DI water & dry.
2381	Yes	Yes, the previously used test vessel(s) were pre-treated	All test vessels are pretreated by 5% Nitric acid for 4 hours and then rinsed with deionized water and dry.
2382	Yes	Yes, the previously used test vessel(s) were pre-treated	the vessel and holder shall be pre-treated by being stored in a solution of dilute nitric acid for at least 4h.After acid cleaning,rinse the vessel and holder with D.I. water and dry.

lab	ISO17043 accredited	pre-cleaning of test vessel	cleaning solution
2385	Yes	No, new/disposable test vessel(s) were used	
2390	Yes	No, new/disposable test vessel(s) were used	
2406	Yes	No, new/disposable test vessel(s) were used	
2415	Yes	No, new/disposable test vessel(s) were used	
2426	-----	-----	
2429	Yes	Yes, the previously used test vessel(s) were pre-treated	Pre-treated by being stored in a solution of dilute nitric acid for at least 4 hours. After acid treatment, rinse the vessel with Milli-Q water and dry.
2442	Yes	No, new/disposable test vessel(s) were used	
2459	Yes	No, new/disposable test vessel(s) were used	
2462	Yes	No, new/disposable test vessel(s) were used	
2475	Yes	---	HNO3 5% during 4H; After acid cleaning, rinse the vessel with deionised water and dry
2489	Yes	No, new/disposable test vessel(s) were used	
2492	Yes	No, new/disposable test vessel(s) were used	
2500	Yes	Yes, the previously used test vessel(s) were pre-treated	2mol/L HNO3 then DI Water
2504	No	No, new/disposable test vessel(s) were used	
2511	Yes	---	
2582	---	---	
2590	Yes	No, new/disposable test vessel(s) were used	
2591	Yes	No, the previously used test vessel(s) were not pre-treated	
2624	No	No, new/disposable test vessel(s) were used	
2629	Yes	No, new/disposable test vessel(s) were used	
2650	Yes	No, new/disposable test vessel(s) were used	
2652	---	---	
2674	Yes	Yes, the previously used test vessel(s) were pre-treated	5%HNO3 steep 4 hours
2701	Yes	No, new/disposable test vessel(s) were used	
2720	Yes	Yes, the previously used test vessel(s) were pre-treated	Soak in 5% nitric acid for 4 hours
2737	Yes	Yes, the previously used test vessel(s) were pre-treated	Lids and vessels are stored in a solution of nitric acid at least 4 hour
2743	Yes	Other	
2818	Yes	No, new/disposable test vessel(s) were used	
2829	Yes	No, new/disposable test vessel(s) were used	
2864	Yes	No, new/disposable test vessel(s) were used	
2910	Yes	No, new/disposable test vessel(s) were used	
2953	---	---	
2959	No	Yes, the previously used test vessel(s) were pre-treated	soak in 5% nitric acid for 24h
2969	Yes	Yes, the previously used test vessel(s) were pre-treated	Wash the vessels with detergent,then soak with 50% HNO3 overnight. Wash with DI water, then dry the vessels with oven.
3100	Yes	Yes, the previously used test vessel(s) were pre-treated	The vessels shall be pre-treatment by being stored in a solution of 5% nitric acid for at least 4 hours,After acid treatment,rinse the Vessels with Grade 1 water and dry.
3110	---	---	
3116	Yes	Yes, the previously used test vessel(s) were pre-treated	Soaked the vessels in 5% nitric acid solution for 4 hours and then rinsed with deionized water.
3118	Yes	No, new/disposable test vessel(s) were used	
3146	Yes	No, new/disposable test vessel(s) were used	
3153	Yes	Other	
3172	Yes	No, new/disposable test vessel(s) were used	
3176	Yes	No, new/disposable test vessel(s) were used	
3182	No	Yes, the previously used test vessel(s) were pre-treated	Soak with 10% HNO3
3185	Yes	Yes, the previously used test vessel(s) were pre-treated	General glassware, are pre-treated by being stored in a solution of dilute nitric acid for at least 4 hours. After acid treatment, rinse the vessel with Milli-Q water and dry.
3210	Yes	No, the previously used test vessel(s) were not pre-treated	
3218	Yes	No, new/disposable test vessel(s) were used	
3220	Yes	Yes, the previously used test vessel(s) were pre-treated	Place the vessel in 5% of HNO3 for 4 hours, after 4 hrs rinse the vessel with distilled water
3228	Yes	No, new/disposable test vessel(s) were used	
3230	Yes	No, new/disposable test vessel(s) were used	

APPENDIX 4

Detailed description on how object was measured for sample #21611

lab	Please, describe as detailed as possible how you have measured and calculated the surface of the object
110	
210	
339	
348	Surface of one link in the chain was calculated by approximation to a cylinder (lateral area). Total surface was calculated as ten times this area (ten links). Dimensional measurements were made with caliper.
623	digital caliper used to measured surface area and calculated by formulation of cylinder multiply by 10 link of chains
840	
841	Determinate Total surface area on 1 link, then multiply by the required number of links.
2102	1 link is opened and the area is calculated using the formulas for a cilinder
2115	
2121	- Flattening a link : it becomes an ellipse - Perimeter (P) of the ellipse was calculated - Section (S) of the ellipse was calculated - Area (A) of the ellipse was calculated : A = P x S for one link - For the 17 links : P x S x 17
2129	
2132	Measure the diameter (R) and length (L) of one link with a digital caliper. Calculate the surface area by 10 x π x R x L.
2139	I measured it with calipers.
2165	Suppose one link tube a ellipse, The area of the ellipse is obtained by multiplying the cross section and the circumference of the ellipse, Then calculate the total area of 10 ellipses.
2184	
2201	S Cylinder : 3.14*0.42mm*8.35mm =11.01198mm2 S Total: S Cylinder*10 =110.1198mm2
2215	S={3.14*0.44*[(1.70-0.44)*3.14+2*(3.42-1.70)]}*10=102.2
2232	Assume it is Cylinder
2236	Link was cut using a razor blade and straightened using tweezers and then a caliper was used to measure radius and L. Area was calculated using A = 2*pi*r*l for an individual link and then multiplied by 10 to determine total area.
2238	After a ring is straightened, the cylindrical area is measured with a vernier caliper.Multiply by 10 to get the total area.
2241	Person 1：0.45*π*8.16*10=115.3592 Person 2：0.43*π*8.17*10=114.7938 Person 3：0.45*π*8.12*10=110.3673 Average：113.51 mm2
2247	Lab straighten one link then calculated its area as cylinder & multiplied it by 10.
2250	chain link bent up, measure with a caliper and calculated as cylinder mantle
2255	By using Slide calipers
2256	Calculate the area of the individual chains and multiply it by 10, the area of a single chain is the diameter of the chain times its length and PI.
2266	measurement carried out with a caliper on a flattened link + mathematical formula for the surface of a geometric shape
2271	
2289	Remove a link from the long chain, and straigten it into a cylider. Then calculate the area of the cylinder. The final result is the area of one cylinder multiplied 10.
2290	
2293	We did separate one link and made the link straight then consider it as a cylinder. After that, we apply the equation of cylinder surface area and multiply by 10.
2310	We calculate the surface area using by vernier caliper
2311	10 x (area of cylinder)
2330	Used digital caliper
2347	
2350	Cut one of the chain and unfold this. And calculate this surface area. 1 link of the chain's surface area: 10.844mm^2 10 links total surface area: 10.844mm^2 * 10= 108.44mm^2
2352	
2357	
2363	
2365	πDl*n
2366	
2369	
2370	First calculate the area of a small circle and then multiply by 10. Area of a small circle: 2 Cylinder area
2374	0.44mm*¦Ð*7.98mm*10links
2375	caliper meter
2377	
2378	
2379	measured by Vernier caliper.
2380	One part of the chain consider as a cylinder. We used the formula, Area of the cylinder=2πrl. Here r=radius of the cylinder & l=length of the cylinder. Total Area=One part area of the cylinderX10.
2381	First of all we consider one link. We consider it as a cylinder & measure length & diameter and then measure the area of a cylinder. Finally multiply by ten.
2382	S=0.42*3.14*8.21*10=108.3(mm2) s-area(mm2) d-the diameter of the ring(cm) c-the perimeter of the ring(cm) n-quantity
2385	Surface was calculated for one link and afterwards multiplicated by 10. The inner and outer dimensions of a single link were measured. Furthermore, the diameter of the link was determined. Then from these values the area was calculated.
2390	Separate one link from chain than make it straight and considered as cylinder and applied formula of cylinder that is πld (3.142*Length*diameter). Finally this value multiply by 10 followed by iis instruction.
2406	Calipers is used to calculate the surface area of 1 link, then the total surface area is 10 times of the surface area of one link.
2415	Consider a link contains 2 straight rods, length 1.5mm/each and two half of a circle with diameter of 1.7mm. Multiply the length of the link with circumference of the wire with a diameter of 0.42mm. Area of 10 links = 10 links * (1.5mm*2 + 3.14*1.7mm) * 3.14*0.42mm = 109.96mm2
2426	By vernier calliper

lab	Please, describe as detailed as possible how you have measured and calculated the surface of the object
2429	Take one of the chains, cut and straighten it, measure its diameter and length, calculate the surface area of one chain, and multiply it by 10 times.
2442	Separate the 10 parts of the chain and measured each surface with a digital caliper.
2459	Surface area of chain is calculated by using Vernier Caliper with 2πr2 + 2πrh
2462	
2475	
2489	We considered as cylinder and used the formula 2πrl. One loop calculated & converted for 10 links
2492	we treat as each link identically, first of all , measure the length and width of the link as well as the thickness. And then multiply each others and accumulated to give the total surface area.
2500	Remove a link ring from the chain, make it into a straight shape with a tool .Calculate the area as the Cylinder similarly;Use the vernier caliper to determine the diameter and length.
2504	change from ring shape to cylinder shape. Calculate 1 cylinder per 1 ring then multiply 10.
2511	
2582	
2590	I used the following geometrical figures: cylinder and bull
2591	We measure the surface of the piece with a vernier caliper with a precision of 0.01mm.
2624	MEASURED SINGLE ELEMENT BY DIGITAL CALIPER AND MULTIPLIED BY 10
2629	Sample area of one link x number of link Sample area of one link: 2 x Area of side + 2 x Area of end
2650	We have taken all 10 links. We have stretched the chain and with a vernier caliper we have measured the rectangle formed.
2652	
2674	measured by vernier caliper calculated by calculator
2701	
2720	Take a chain, straighten it, take it as a cylinder, calculate the area, measure it with vernier caliper, calculate the area of a chain, and the final area is 10 times of the chain
2737	One link is projected onto paper to form an ellipse, with a long half axis a=1.5mm, short half axis b=0.7mm, and the formula l=2πb+4(a-b) is used to calculate its perimeter; Measured the link diameter d=0.46mm,The surface area of one link is s' =π* D * l,total area of 10 links is 10*s'
2743	
2818	S=（2π*r*L+2π*r*r)*10
2829	
2864	Straighten the sample and measure the length and diameter.
2910	S= π*d*l*10pcs
2953	
2959	Truncate a link with the approprate tool and carefully expand it into a cylinder. Calculate using the calculation formula S'=π*d*l for the cylinder's surface area, where d=0.45mm and l=8.27mm. And the total area of 10 links is S=10*S'.
2969	Cut each of the links (total 10PCS), Use calliper to test the diameter and the length of the link, caculate the surface area of each link.
3100	Each link is calculated as a cylinder,then added up the areas of the 10 links.
3110	
3116	
3118	1. Cut one ring of chain, and then shape it straight like a tube. 2. Measured in diameter and length. 3. Calculate the area such as the area of the tube blanket. 4. Calculate the total of all rings
3146	Calculation of a single chain link: This was bent and calculated as a cylinder. From this cylinder, the sheath surface was used as a surface result. This result was multiplied by the number of chain links (10).
3153	Measure diameter of the link with a digital caliper. Calculate the circumference of the link. Measure perimeter of the link. Calculate the surface area of the link by calculating the product of perimeter and circumference of the link. Calculate the total surface area by multiplying the product by 10
3172	Digital Caliper
3176	
3182	(2*3.14*r*h)*21. Reply op check mail (30-6-21): Calculate (2(3.14)(0.215)(7.92))*10 = 106.936
3185	Take a small ring, straighten it, and then calculate it according to the formula for calculating the side area of the cylinder
3210	
3218	Perimeter:C=8.76mm diameter£ºb=0.41mm area=10*¥ð*C*b=10*3.14*8.76*0.41=112.77mm²©÷
3220	First c+D38alculate the surface area of single link of metal chain by using the formula of surface area of cylindrical rod ($2\pi rh + 2\pi r^2$) then multiple the surface area of single link to 10 times to calculate the surface area of 10 links
3228	let one piece of the chain on a plane and then it can be as a oval. calculate the circumference of oval. use the diameter of wire and circumference calculate the area of one piece.
3230	Area of 10 links = 10 x {[Area of large ellipse – Area of small ellipse] x 2 + Surface area of cylinder (inner + outer side)} Area of 10 links = 10 x {[4.4180 – 0.3835] x 2 + (2.1614 + 0.8294)} = 110.598 mm2 ←, Measurement was done using a vernier calliper

APPENDIX 5

Number of participants per country

4 labs in	BANGLADESH
1 lab in	CAMBODIA
5 labs in	FRANCE
4 labs in	GERMANY
1 lab in	GUATEMALA
8 labs in	HONG KONG
5 labs in	INDIA
2 labs in	INDONESIA
7 labs in	ITALY
1 lab in	MAURITIUS
1 lab in	MOROCCO
33 labs in	P.R. of CHINA
3 labs in	PAKISTAN
1 lab in	SINGAPORE
3 labs in	SOUTH KOREA
3 labs in	SPAIN
1 lab in	SRI LANKA
2 labs in	TAIWAN
3 labs in	THAILAND
1 lab in	THE NETHERLANDS
1 lab in	TUNISIA
2 labs in	TURKEY
2 labs in	U.S.A.
5 labs in	VIETNAM

APPENDIX 6

Abbreviations

C	= final test result after checking of first reported suspect test 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 outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from the statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported

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