Results of Proficiency Test
Nickel Release
June 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Nickel has always been used in various applications, as a pure metal, as a plated substance on another metal or as an alloy. Nickel applications usually do not give problems, but when Nickel comes into prolonged and direct contact with the human skin, sensitization can occur. When a person becomes sensitive to Nickel, even the smallest amounts can provoke an allergic reaction. By this, Nickel is the most frequent cause of contact allergy in Europe. Both the contact itself (sometimes enhanced by damaged skin) and skin conditions as sweat can cause the body to be exposed 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 (ear rings), 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 every year. During the annual proficiency testing program 2018/2019, it was decided to continue the proficiency test for the analysis of Nickel release. In this interlaboratory study 129 laboratories in 30 different countries registered for participation. See appendix 5 for the number of participants per country. In this report, the test results of the 2019 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 #19555 positive on Nickel release and one piece of a metallic chain consisting of 6 links labelled #19556 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. Also, some analytical details were asked.

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 a 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

The batch of Nickel containing material 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 not plated or coated. The dimensions of each item were approximately 2x2x0.2cm and the hole had a diameter of approximately 5mm. The sample was labelled #19555 and was packed per three items in a plastic bag. Twenty-four stratified randomly selected (sub)samples were tested using EN1811:11+A1:15 and single test results were averaged per three to check the homogeneity of the batch. The test results of the homogeneity tests are shown in table 1.

	Nickel Release (µg/cm^2/week) averaged per 3 items
sample #19555-1	2.12
sample #19555-2	2.21
sample #19555-3	2.25
sample #19555-4	2.18
sample #19555-5	2.18
sample #19555-6	2.22
sample #19555-7	2.16
sample #19555-8	2.16

Table 1: homogeneity test results of subsamples #19555

From the above test results the repeatability was calculated and compared with 0.3 times the target reproducibility, estimated from the Horwitz equation, in agreement with the procedure of ISO13528, Annex B2, in the next table.

	Nickel release (µg/cm^2/week)
r (observed)	0.11
reference method	Horwitz *)
0.3 x R (reference method)	0.26

Table 2: evaluation of the repeatability of subsamples #19555

*) The Horwitz formula is converted to µg/cm^2/week unit instead of a concentration

The calculated repeatability was in agreement with 0.3 times the target reproducibility estimated from the Horwitz equation. Therefore, homogeneity of the (sub)samples #19555 was assumed.

Surface Determination
A batch of a metal chain was obtained from a local supplier. From this batch, 150 plastic bags were filled each with six links of the chain. The samples were labelled #19556. No homogeneity tests were done because only surface determination has been requested for this sample. However, each sample was weighed in advance to ensure no large differences in surfaces.

To each of the participating laboratories three items of sample #19555 and one sample #19556 were sent on May 29, 2019.

2.5 ANALYZES

The participants were requested to determine Nickel Release on sample #19555 and to determine surface only on sample #19556, applying the analysis procedure that is routinely used in the laboratory. It was also requested 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 appropriate reference test methods 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 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 data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's 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'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. The Kernel Density Graph 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 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, 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 target reproducibility by division with 2.8. In case no literature reproducibility was available, other target values are used.
In some cases, a reproducibility based on former iis proficiency tests could be used.

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-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. 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

During the execution of this proficiency test no problems were encountered. Three participants reported test results after the reporting deadline and two other participants did not report any test results at all. In total 127 participants reported 251 test results for Nickel Release and Surface Determination. Observed were 7 outlying test results, which is 2.8%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.
Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER SAMPLE

In this section, the reported test results are discussed per sample. All statistical results reported on the samples are summarised in appendix 1. The abbreviations used in these tables are listed 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 and ranges from 54% at 0.3 µg Ni/cm^2/week up to 32% at 10 µg Ni/cm^2/week.

Sample #19555:

Nickel Release: This determination may be problematic at the low concentration level of 1.19 µg/cm^2/week. Five statistical outliers were observed and nine other test results were excluded on the basis of the surface determination of the (sub)samples.
The calculated reproducibility after rejection of the suspect data is not in agreement with the target reproducibility estimated from the Horwitz equation. The low Nickel Release level may (partly) explain the relatively large variation.

Sample #19556:

Surface Determination: This determination on the chain may not be problematic. Two statistical outliers were observed in the reported range of 6.61-11.03cm^2. No official test method exists for Surface Determination. Therefore, no z-scores were calculated. The variation for this sample of 7% is in line with the observed variation in previous PT’s in which the Surface Determination was evaluated (4.9-13%), but is large compared to the variation of the Surface Determination of the much simpler shaped sample #19555 (1.1%).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the calculated reproducibility (2.8 * standard deviation) as found for the group of participating laboratories and the target reproducibility estimated from the Horwitz equation in the next table.

Parameter	unit	n	average	2.8 * sd	R (target)
Nickel Release	µg/cm^2/week	112	1.19	0.99	0.53
Surface	cm^2	107	9.47	0.28	n.a.

Table 3: reproducibilities of test results on sample #19555

From table 3 it can be concluded, without further statistical calculations, that the group of participating laboratories had problems with the analyzis of Nickel Release when compared to the Horwitz target reproducibility.

Parameter	unit	n	average	2.8 * sd	R (target)
Surface Determination	cm^2	123	8.96	1.74	n.a.

Table 4: reproducibility of test results on sample #19556

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2019 WITH PREVIOUS PTS

	June 2019	May 2018	May 2017	May 2016	May 2015
Number of reporting laboratories	127	113	122	125	123
Number of test results	126	112	122	124	119
Number of statistical outliers	5	4	14	8	11
Percentage outliers	4.0%	3.6%	11%	6.5%	9.8%

Table 5: comparison with previous proficiency tests (Nickel Release determination only)

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.
In table 6 the observed uncertainties in this PT are compared with the uncertainties as observed in the previous PTs.

	June 2019	May 2018	May 2017	May 2016	2015-2014
Nickel Release	30%	44%	26%	18%	27-31%
Surface Determination	1.1 - 7%	1.3 - 13%	1.3 - 6.7%	2.3 - 4.9%	1.7-10%

Table 6: comparison of uncertainties (relative in %) of current PT with previous PTs

The uncertainty of the Nickel Release determination is in line with the average uncertainty from the previous years.
Also the uncertainty of the Surface Determinations is in line with the average uncertainty of previous samples for Surface Determination.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For the three (sub)samples #19555 (Nickel Release) the following can be summarized from the various analytical details provided by the participants (see appendix 2 and 3):

- a majority of 95% of the reporting participants used all three plates for the Nickel Release determination.
- about 50% of the reporting participants have done a pre-treatment of the test vessels, while 8% of the reporting participants have used new or disposable test vessels. Remarkably, 31% of the reporting participants have not done any pre-treatment and 9% did not answer this question, see appendix 3.
- a majority of around 70% of the reporting participants used a ratio of approximately 1mL test solution per cm^2 sample surface area. Surprisingly a few participants reported to have used between two and ten times as much volume of test solution than sample area.
- in total 117 laboratories (92%) reported the average surface area, see appendix 2. The average surface area varied from 5.6 to 16.7cm^2.
- in total 105 laboratories (83%) reported to be accredited for this test in accordance with ISO/IEC17025.

For sample #19556 (Surface Determination) only one question was requested: a detailed description on how the surface area was measured and calculated which was answered by 76% of the reporting participants. A divers variety of methods was given, see appendix 4.

5 DISCUSSION

Possible effects of the various analytical details on Nickel Release were investigated.

The range for the surface area of (sub)samples #19555 narrowed between 9.2 to 9.8cm^2, after exclusion of ten statistically outlying data. The observed RSD of 1.1% is somewhat lower than in the previous PT (1.3%). In this PT the overall RSD for Nickel Release for sample #19555 is 30%. This is the sum of the variation in contact surface determination and the variation in the Nickel determination. It can be concluded that the variation in the surface determination of this simple squared object does not affect the overall variation of the Nickel Release determination.

The variation on the Nickel Release results for the individual items was huge; from 0.4 up to 149 RSD (%). Only 28 participants had an RSD in agreement with the target repeatability standard deviation of 4%, estimated from EN1811:11+A1:15 (33.3% / 2.8 / 3). However, when evaluating the test results of only those 28 participants the average and variation are not significant different from the total group, see appendix 1.

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.

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 9-10mL per item. Not all participants used this ratio. However, when evaluating the test results of only those participants that used a 1:1 ratio for test solution: surface area than again the average and variation are not significant different from the total group, see appendix 1.

6 CONCLUSION

It can be concluded that a large group of the participants 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.

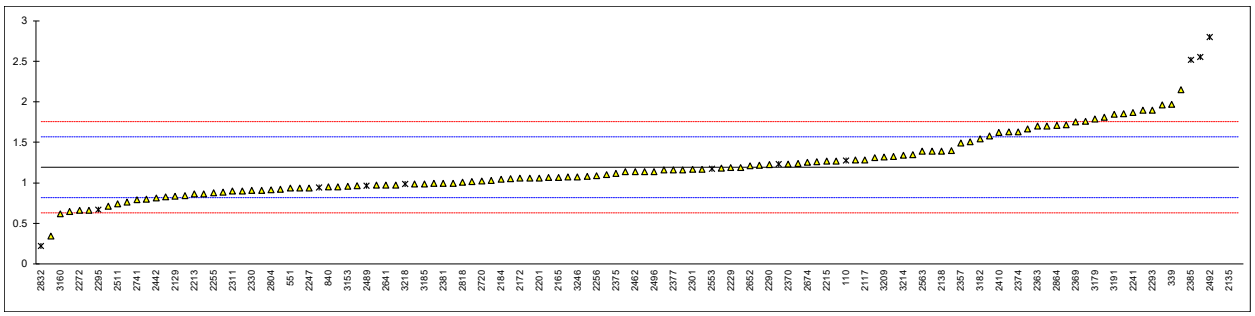
APPENDIX 1

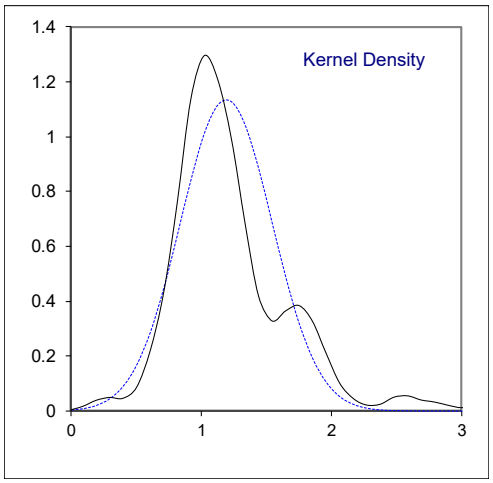
Determination of Nickel Release on sample #19555; average result of three replicates in µg/cm²/week

lab	method	value	mark	z(targ)	remarks
110	In house	1.2729	ex	0.43	outlier in sample surface, see appendix 2
210		-----		-----	
230	EN1811	1.965		4.12	
339	EN1811 + A1	1.97		4.15	
348	EN1811 + A1	1.075		-0.63	
362	EN1811 + A1	0.943	ex	-1.33	outlier in sample surface, see appendix 2
523	EN1811 + A1	1.7615		3.04	
551	EN1811	0.9354		-1.37	
623	EN1811 + A1	1.81	C	3.30	first reported 2.38
840	EN1811 + AC	0.95		-1.29	
2102	EN1811	1.348		0.83	
2115	EN1811	0.765		-2.28	
2117	EN1811 + A1	1.2800		0.47	
2129	EN1811	0.837		-1.90	
2132	EN1811	0.9368		-1.36	
2135	EN1811 + A1	4.697	R(0.01)	18.71	
2137	EN1811 + A1	2.551	R(0.05)	7.25	
2138	EN1811	1.394		1.08	
2139	EN1811 + A1	0.867		-1.74	
2165	EN1811	1.070		-0.65	
2172	EN1811 + A1	1.061		-0.70	
2184	EN1811 + A1	1.042		-0.80	
2201	EN1811 + A1	1.062		-0.70	
2213	EN1811	0.866		-1.74	
2215	EN1811 + A1	1.266		0.39	
2216	EN1811	1.665		2.52	
2229	EN1811 + A1	1.19		-0.01	
2232	EN1811 + A1	1.163		-0.16	
2238	EN1811 + AC	1.26		0.36	
2241	EN1811	1.868		3.61	
2247	EN1811	0.94		-1.35	
2250	EN1811 + A1	3.20	C,R(0.01)	10.72	first reported 2.687
2255	EN1811	0.878		-1.68	
2256	EN1811 + A1	1.088		-0.56	
2265	EN1811	2.15		5.11	
2272	EN1811 + A1	0.66		-2.84	
2289	EN1811 + A1	1.061		-0.70	
2290	EN1811 + A1	1.225		0.17	
2293	In house	1.9002		3.78	
2295	EN1811	0.67	ex	-2.79	outlier in sample surface, see appendix 2
2301	EN1811	1.165		-0.15	
2310	EN1811 + AC	1.23	ex	0.20	outlier in sample surface, see appendix 2
2311	EN1811 + AC	0.9017		-1.55	
2330	EN1811	0.9099		-1.51	
2347	EN1811 + A1	1.90		3.78	
2350	EN1811 + A1	1.241		0.26	
2352	EN1811	1.271		0.42	
2357	EN1811	1.491		1.59	
2363	EN1811 + A1	1.70	C	2.71	first reported 2.205
2365	EN1811 + A1	1.719		2.81	
2366	EN1811 + A1	1.850		3.51	
2369	EN1811 + A1	1.752	C	2.99	first reported 2.109
2370	EN1811	1.23		0.20	
2374	EN1811	1.63	C	2.34	first reported 2.06
2375	EN1811 + A1	1.12		-0.39	
2377	EN1811 + A1	1.16		-0.17	
2378	EN1811	1.310		0.63	
2379	EN1811	1.504		1.66	
2380	EN1811	1.069		-0.66	
2381	EN1811	0.995		-1.05	
2382	EN1811 + A1	1.28		0.47	
2385	EN1811	2.52	R(0.05)	7.09	
2390	EN1811 + A1	0.65		-2.90	
2406	EN1811 + A1	1.08		-0.60	
2410		1.62		2.28	
2415	EN1811 + A1	1.40		1.11	
2429	EN1811 + A1	0.998		-1.04	
2442	EN1811 + A1	0.817		-2.00	
2459	EN1811	0.976		-1.16	
2462	EN1811	1.14		-0.28	
2475	EN1811 + A1	1.033		-0.85	
2482	EN1811 + A1	1.392		1.07	
2489	EN1811	0.966	ex	-1.21	outlier in sample surface, see appendix 2
2492	EN1811	2.80	C,R(0.05)	8.58	first reported 2.55
2495	EN1811	1.191		-0.01	

lab	method	value	mark	z(targ)	remarks
2496	EN1811 + A1	1.142		-0.27	
2497	EN1811 + AC	26.06	ex	132.75	outlier in sample surface, see appendix 2
2500	EN1811	1.1032		-0.48	
2511	EN1811	0.740		-2.41	
2514	EN1811	0.924		-1.43	
2522	EN1811 + A1	1.14		-0.28	
2532	EN1811	0.992		-1.07	
2553	EN1811	1.176	ex	-0.09	outlier in sample surface, see appendix 2
2560	EN1811	0.714		-2.55	
2563	EN1811	1.39		1.06	
2582	EN1811	0.9527		-1.28	
2590	EN1811	0.8001		-2.09	
2624	EN1811 + A1	0.66		-2.84	
2629	EN1811	1.326		0.71	
2641	EN1811	0.971		-1.18	
2652	EN1811	1.2118		0.10	
2657	EN1811 + A1	1.138		-0.29	
2674	EN1811	1.257		0.35	
2703	EN1811	0.344	C	-4.53	first reported 0.47027
2705	EN1811 + AC	1.182		-0.06	
2719	In house	1.7		2.71	
2720	EN1811 + A1	1.027		-0.88	
2741	EN1811 + A1	0.79		-2.15	
2758	EN1811	1.627		2.32	
2804	EN1811	0.9181		-1.46	
2818	EN1811 + A1	1.011		-0.97	
2829	EN1811 + AC	0.903		-1.54	
2832	EN1811 + A1	0.22	ex	-5.19	outlier in sample surface, see appendix 2
2833		-----		-----	
2858	EN1811	0.829		-1.94	
2864	EN1811 + A1	1.7079		2.75	
2878	EN1811	0.9696		-1.19	
3100	EN1811 + A1	0.963		-1.22	
3116	EN1811 + A1	1.1577		-0.19	
3118	EN1811	1.5784		2.06	
3146	EN1811 + A1	0.842		-1.87	
3150		-----		-----	
3153	EN1811 + A1	0.9604		-1.24	
3154	EN1811	1.053		-0.74	
3160	EN1811 + A1	0.62	C	-3.06	first reported 36.23
3172	EN1811	1.22		0.15	
3179	EN1811 + A1	1.79		3.19	
3182	EN1811	1.540		1.86	
3185	EN1811 + A1	0.989		-1.09	
3191	EN1811 + A1	1.843		3.47	
3197	EN1811 + A1	0.91		-1.51	
3209	EN1811	1.32		0.68	
3210	EN1811	0.89		-1.61	
3214	EN1811 + A1	1.339		0.78	
3218	EN1811	0.985	ex	-1.11	outlier in sample surface, see appendix 2
3228	EN1811 + A1	1.17		-0.12	
3237	EN1811	0.9883		-1.09	
3246	EN1811 + A1	1.077		-0.62	
3248	EN1811	1.018		-0.93	

				only with ratio 1:1		only RSD <4% between replicates	
	normality	OK		OK		OK	
	n	112		87		28	
	outliers	5 +9ex		2		1	
	mean (n)	1.192		1.193		1.084	
	st.dev. (n)	0.3517	RSD=30%	0.3316	RSD=28%	0.3003	RSD=28%
	R(calc.)	0.985		0.928		0.841	
	st.dev.(Horwitz)	0.1873		0.1874		0.1728	
	R(Horwitz)	0.525		0.525		0.484	
Compare							
	R(EN1811:11+A1:15)	0.397		0.397		0.361	


3
2.5
2
1.5
1
0.5
0
2832 3160 2272 2295 2511 2741 2442 2129 2213 2255 2311 2330 2804 551 2247 840 3153 2489 2641 3218 3185 2381 2818 2720 2184 2172 2201 2165 3246 2256 2375 2462 2496 2377 2301 2553 2229 2652 2290 2370 2674 2215 110 2117 3209 3214 2563 2138 2357 3182 2410 2374 2363 2864 2369 3179 3191 2241 2293 339 2385 2492 2135

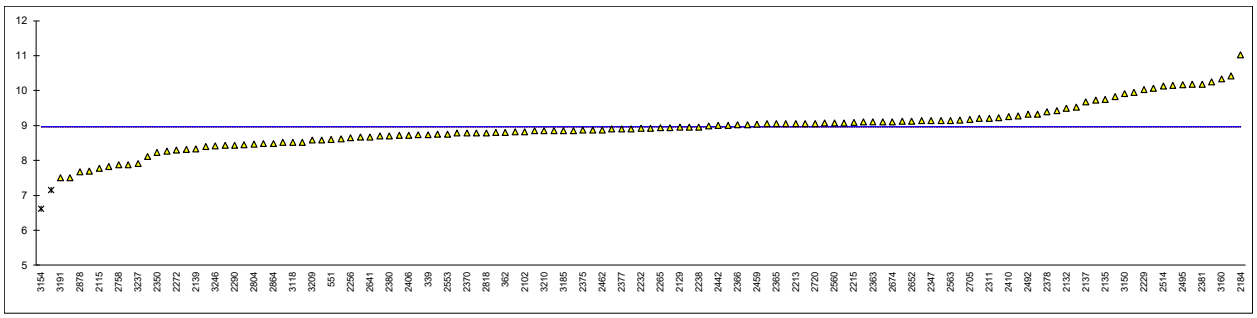

Kernel Density
1.4
1.2
1
0.8
0.6
0.4
0.2
0
0
1
2
3

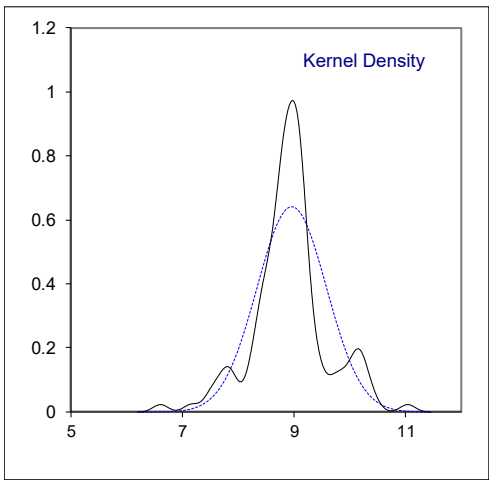
Determination of Surface on sample #19556; results in cm^2

lab	method	value	mark	z(targ)	remarks
110	see appendix 4	9.333		-----	
210		-----		-----	
230	see appendix 4	8.517		-----	
339	see appendix 4	8.74		-----	
348	see appendix 4	8.585		-----	
362	see appendix 4	8.809		-----	
523	see appendix 4	8.9196		-----	
551	see appendix 4	8.60		-----	
623	see appendix 4	10.42		-----	
840		-----		-----	
2102	see appendix 4	8.826		-----	
2115	see appendix 4	7.78		-----	1 ring= 1.296cm^2
2117	see appendix 4	8.2597		-----	
2129	see appendix 4	8.95		-----	
2132	see appendix 4	9.489		-----	
2135	see appendix 4	9.75		-----	
2137	see appendix 4	9.683	C	-----	first reported 21.85
2138	see appendix 4	7.872		-----	
2139	see appendix 4	8.33		-----	
2165	see appendix 4	8.720		-----	
2172	see appendix 4	8.898		-----	
2184	see appendix 4	11.030		-----	
2201	see appendix 4	8.62		-----	
2213	see appendix 4	9.06		-----	
2215	see appendix 4	9.095		-----	
2216		-----		-----	
2229	see appendix 4	10.04		-----	
2232	see appendix 4	8.913		-----	
2238	see appendix 4	8.96		-----	
2241	see appendix 4	8.52		-----	
2247	see appendix 4	9.05		-----	
2250	see appendix 4	9.06	C	-----	first reported 1.51 per chain
2255	see appendix 4	8.901		-----	
2256	see appendix 4	8.6455		-----	
2265	see appendix 4	8.938		-----	
2272	see appendix 4	8.294		-----	
2289	see appendix 4	9.12		-----	
2290	see appendix 4	8.44		-----	
2293	see appendix 4	9.1		-----	
2295	see appendix 4	8.7		-----	
2301	see appendix 4	8.779		-----	
2310	see appendix 4	8.31		-----	
2311	see appendix 4	9.206		-----	
2330	see appendix 4	8.956		-----	
2347	see appendix 4	9.14		-----	
2350	see appendix 4	8.238		-----	
2352	see appendix 4	9.271		-----	
2357	see appendix 4	9.14		-----	
2363	see appendix 4	9.10		-----	
2365	see appendix 4	9.055		-----	
2366	see appendix 4	9.02		-----	
2369	see appendix 4	9.14		-----	
2370	see appendix 4	8.78		-----	
2374	see appendix 4	9.1		-----	
2375	see appendix 4	8.87		-----	
2377	see appendix 4	8.90		-----	
2378	see appendix 4	9.391		-----	
2379	see appendix 4	10.249		-----	
2380	see appendix 4	8.70		-----	
2381	see appendix 4	10.191		-----	
2382	see appendix 4	9.152		-----	
2385	see appendix 4	8.4		-----	
2390	see appendix 4	8.94		-----	
2406	see appendix 4	8.725		-----	
2410	see appendix 4	9.26		-----	
2415	see appendix 4	8.43		-----	
2429	see appendix 4	9.23		-----	
2442	see appendix 4	9.0		-----	
2459	see appendix 4	9.046		-----	
2462	see appendix 4	8.874		-----	
2475	see appendix 4	10.07		-----	
2482	see appendix 4	9.42		-----	
2489	see appendix 4	9.06		-----	
2492	see appendix 4	9.318		-----	
2495	see appendix 4	10.17		-----	

lab	method	value	mark	z(targ)	remarks
2496	see appendix 4	8.815		-----	
2497	see appendix 4	7.511		-----	
2500	see appendix 4	8.8709		-----	
2511	see appendix 4	8.86		-----	
2514	see appendix 4	10.13		-----	
2522	see appendix 4	8.12		-----	
2532	see appendix 4	9.066		-----	
2553	see appendix 4	8.76		-----	
2560	see appendix 4	9.068		-----	
2563	see appendix 4	9.14		-----	
2582	see appendix 4	8.4475		-----	
2590	see appendix 4	7.6859		-----	
2624	see appendix 4	10.18		-----	
2629	see appendix 4	7.161	DG(0.05)	-----	
2641	see appendix 4	8.672		-----	
2652	see appendix 4	9.121207		-----	
2657	see appendix 4	10.149		-----	
2674	see appendix 4	9.110		-----	
2703	see appendix 4	9.074562637		-----	
2705	see appendix 4	9.167		-----	
2719	see appendix 4	8.8		-----	
2720	see appendix 4	9.06		-----	
2741	see appendix 4	9.52		-----	
2758	see appendix 4	7.87		-----	
2804	see appendix 4	8.471		-----	
2818	see appendix 4	8.79		-----	
2829	see appendix 4	7.821		-----	
2832	see appendix 4	8.85		-----	
2833		-----		-----	
2858	see appendix 4	9.0		-----	
2864	see appendix 4	8.49		-----	
2878	see appendix 4	7.669		-----	
3100	see appendix 4	8.73		-----	
3116	see appendix 4	9.73		-----	
3118	see appendix 4	8.51833		-----	
3146	see appendix 4	9.200		-----	
3150	see appendix 4	9.91		-----	
3153	see appendix 4	9.83		-----	
3154	see appendix 4	6.61	C,DG(0.05)	-----	first reported 15.43
3160	see appendix 4	10.33		-----	
3172	see appendix 4	8.8601		-----	
3179	see appendix 4	8.9825		-----	
3182	see appendix 4	8.481		-----	
3185	see appendix 4	8.86		-----	
3191	see appendix 4	7.5	C	-----	first reported 125
3197	see appendix 4	8.75		-----	
3209	see appendix 4	8.58		-----	
3210	see appendix 4	8.8546		-----	
3214	see appendix 4	8.783		-----	
3218	see appendix 4	8.670		-----	
3228	see appendix 4	9.02		-----	
3237	see appendix 4	7.91		-----	
3246	see appendix 4	8.41		-----	
3248	see appendix 4	9.943		-----	

	normality	suspect	
	n	123	
	outliers	2	
	mean (n)	8.9572	
	st.dev. (n)	0.62056	RSD=7%
	R(calc.)	1.7376	
	st.dev.(target)	n.a.	
	R(target)	n.a.	


12
11
10
9
8
7
6
5
3154 3191 2878 2115 2758 3237 2350 2272 2139 3246 2290 2804 2864 3118 3209 551 2256 2641 2380 2406 339 2553 2370 2818 362 2102 3210 3185 2375 2462 2377 2232 2265 2129 2238 2442 2366 2459 2365 2213 2720 2560 2215 2363 2674 2652 2347 2563 2705 2311 2410 2492 2378 2132 2137 2135 3150 2229 2514 2495 2381 3160 2184


Kernel Density
1.2
1
0.8
0.6
0.4
0.2
0
5
7
9
11

APPENDIX 2

Determination of Nickel Release and some Analytical Details on subsamples #19555

lab	value plate 1 (µg/cm2/ week)	value plate 2 (µg/cm2 /week)	value plate 3 (µg/cm2 /week)	RSDr (%) calc. by iis	area of sample used in the calculation for Ni Release (cm²)	mark	volume test solution (mL)	ratio test solution vs. sample area (mL/cm²)
110	1.0392	1.3693	1.4103	16.0	7.904	R(0.01)	8.0	1
210	-----	-----	-----	-----	-----		-----	-----
230	1.888	1.914	2.094	5.7	9.55		10	1:10
339	2.17	1.72	2.01	11.6	9.17		9.2	1:1
348	1.187	1.266	0.773	24.6	9.45		25	2.65
362	-----	-----	-----	-----	9.92	R(0.05)	10	1.0
523	1.5914	1.8692	1.8238	8.5	9.45		13	1.38
551	0.6098	0.5676	1.6288	64.2	9.47		15	1.58
623	2.15	2.42	2.57	8.9	9.5		9.5	1:1
840	0.99	0.94	0.92	3.8	9.41		10	1:1
2102	1.151	1.732	1.162	24.6	9.5		9.5	1:1
2115	-----	0.805	0.735	6.4	9.286		12	1:1
2117	0.9503	1.5372	1.3524	23.4	9.55		12	1.26
2129	0.68	1.02	0.81	20.5	9.62		14	1.66
2132	1.0882	0.9906	0.7317	19.7	9.525		9.5	1:0.997
2135	12.79	0.831	0.47	149.3	9.44		40	4
2137	2.579	2.511	2.563	1.4	9.5		10	1:1
2138	1.403	1.376	1.403	1.1	-----		-----	-----
2139	0.689	1.299	0.613	43.4	9.4		15	1:1.5
2165	1.287	1.066	0.848	20.6	9.52		10	1:0.952
2172	1.056	0.9231	1.204	13.2	9.52		10	1:1
2184	1.032	1.190	0.906	13.6	-----		-----	-----
2201	1.119	0.906	1.160	12.8	9.5		9.5	1:1
2213	0.916	0.854	0.830	5.1	9.41		12.5	-----
2215	1.334	1.052	1.411	14.9	9.39		10	1.06
2216	1.515	1.256	2.224	30.1	9.5		9	1
2229	1.34	1.09	1.15	10.9	9.51		9	0.95
2232	1.156	1.160	1.173	0.8	9.457		9.5	1:1
2238	1.011	1.619	1.166	25.0	9.3		9.3	1:1
2241	2.748	1.766	1.089	44.7	9.41		10.5	1:1
2247	1.07	0.91	0.85	12.1	9.52		10	1:1
2250	3.17	3.35	3.08	4.3	9.42		10	1.0616
2255	0.882	0.878	0.875	0.4	9.26		10	1:1
2256	0.941	1.107	1.215	12.7	9.508		10	1:1
2265	2.14	2.95	1.36	37.0	9.46		15	1:1.5
2272	0.74	0.84	0.40	34.9	9.42		10	1:1
2289	0.976	1.490	0.719	37.0	9.7		9.7	1:1
2290	1.470	0.983	1.221	19.9	-----		-----	-----
2293	1.9283	2.1022	1.6702	11.4	9.49		9.5	1:1
2295	0.69	0.68	0.65	3.1	10	R(0.01)	20	2:1
2301	1.123	1.136	1.238	5.4	-----		-----	-----
2310	1.16	1.31	1.21	6.2	8.9	R(0.01)	9	1:1
2311	0.7773	1.0932	0.8346	18.7	9.42		9.5	1:1
2330	0.9384	0.8937	0.8975	2.7	9.4459		10	1:1
2347	1.74	2.01	1.95	7.5	-----		-----	-----
2350	0.647	1.287	1.789	46.1	9.393		10	1:1
2352	1.053	1.281	1.478	16.7	9.42		9.45	-----
2357	1.887	1.336	1.249	23.2	9.47		10	1:1
2363	2.184	2.182	2.250	1.8	9.5		9.5	1:1
2365	1.298	1.939	1.919	21.2	9.51		9.51	1:1
2366	1.770	1.807	1.974	5.9	9.45		9.5	1:1
2369	1.589	1.721	1.947	10.3	9.4321		10	1:1
2370	1.21	1.22	1.26	2.2	9.41		10	1:1
2374	1.72	1.59	1.58	4.8	9.53		15	25:9.53
2375	1.21	1.09	1.05	7.5	9.43		10	1:1
2377	0.93	1.76	0.78	45.6	9.55		10	1:1
2378	1.462	1.311	1.157	11.6	9.47		9.50	-----
2379	1.873	1.610	1.031	28.6	9.43		10	20:1
2380	1.118	1.059	1.030	4.2	9.53		10	1:1
2381	0.983	1.009	0.992	1.3	9.55		10	1:1
2382	1.29	1.28	1.27	0.8	-----		-----	-----
2385	2.52	2.39	2.65	5.2	9.5		12	1:1.26
2390	0.64	0.69	0.61	6.2	9.2		9.2	1:1
2406	1.06	1.11	1.06	2.7	9.54		9.6	1:1
2410	1.58	1.51	1.77	8.3	9.49		12	1.26:1
2415	1.46	1.37	1.38	3.5	9.66		10	1:1
2429	0.934	1.096	0.963	8.7	9.52		9.5	1:1
2442	0.839	0.835	0.778	4.2	9.49		10	10
2459	0.969	0.972	0.986	0.9	9.48		17	2:1
2462	1.13	1.16	1.14	1.3	9.4		10	1.06
2475	0.98	1.07	1.05	4.6	9.48		9.48	1
2482	1.010	1.651	1.515	24.3	9.28		9.28	1:1

lab	value plate 1 (µg/cm2/ week)	value plate 2 (µg/cm2 /week)	value plate 3 (µg/cm2 /week)	RSDr (%) calc. by iis	area of sample used in the calculation for Ni Release (cm²)	mark	volume test solution (mL)	ratio test solution vs. sample area (mL/cm²)
2489	1.045	0.92	0.93	7.2	9.96	R(0.01)	10	10:5-10
2492	2.987	2.615	2.048	18.5	9.40		12	1.28
2495	0.685	1.159	1.728	43.9	9.48		10	1
2496	1.400	0.592	1.435	41.7	-----		10	10:9.3
2497	12.88	1.09	64.31	128.8	9.06	R(0.05)	50	5
2500	1.1236	1.1606	1.0254	6.3	9.56		10	1:1
2511	0.790	0.660	0.760	9.2	-----		-----	-----
2514	0.925	1.022	0.824	10.7	9.6		9.6	1:1
2522	1.12	1.23	1.08	6.8	9.43		10	1:1
2532	1.28	0.956	0.74	27.4	9.51		20	25:9.5
2553	1.182	1.166	1.179	0.7	5.574	R(0.01)	15	1:3
2560	0.749	0.680	0.712	4.8	9.496		12	1.3:1
2563	1.41	1.52	1.23	10.6	9.456		20	2.1
2582	0.9696	0.9459	0.9427	1.5	9.4257		9	10:5-10
2590	0.8295	0.7933	0.7776	3.3	9.43		20	2:1
2624	0.79	0.39	0.80	35.4	9.41		13	1.4
2629	1.448	1.182	1.349	10.1	9.6		25	5
2641	0.966	0.960	0.987	1.5	9.42		10	1
2652	1.0833	1.2188	1.3333	10.3	9.6		9.6	1:1
2657	1.338	0.946	1.129	17.2	9.259		9.0	0.972
2674	1.097	1.425	1.250	13.1	9.58		15	1.57:1
2703	0.34869	-----	0.33848	2.1	9.75		8	1:1.2
2705	1.527	1.053	0.968	25.5	9.5		100	10
2719	1.7	-----	-----	-----	-----		-----	-----
2720	1.029	1.055	0.998	2.8	9.8		9.8	1:1
2741	0.689	0.833	0.849	11.1	9.5		10	1.1
2758	1.717	1.479	1.685	7.9	9.43		11	1.2
2804	0.9892	0.8584	0.9070	7.2	9.5		9.5	1:1
2818	0.887	1.208	0.937	17.1	9.54		9.54	1.0
2829	0.715	0.791	1.204	29.1	9.59		10	1:1
2832	0.14	0.36	0.15	57.3	9	R(0.01)	11	1.2
2833	-----	-----	-----	-----	-----		-----	-----
2858	0.724	0.882	0.882	11.0	9.5		10	1:1
2864	1.6577	1.7546	1.7115	2.8	9.29		9.3	1:1
2878	0.8547	0.9684	1.0857	11.9	9.38		10	1:1
3100	1.116	0.832	0.942	14.9	9.5		9.5	1:1
3116	0.7410	0.9925	1.745	45.1	9.36		10	1:1
3118	1.5742	1.5921	1.5687	0.8	9.27		10	1.08
3146	0.821	0.863	-----	3.5	9.5		9.5	1:1
3150	0.788	1.89	12.13	126.7	16.74	R(0.01)	17	1:1
3153	0.9691	0.9953	0.9167	4.2	9.534		9.5	1:1
3154	1.54	0.85	0.77	40.2	9.52		50	5.25
3160	0.78	0.46	0.62	25.8	9.5015		10	1.05
3172	-----	-----	-----	-----	9.257		9.25	1:1
3179	0.96	2.17	2.23	40.1	9.45		50	5.3
3182	1.408	1.661	1.550	8.2	9.47		9.47	1:1
3185	1.03	1.01	0.928	5.5	9.40		10	1:1
3191	1.332	1.987	2.209	24.7	9.45		9.45	1:1
3197	0.91	0.96	0.86	5.5	9.5		9.5	1:1
3209	1.33	1.31	1.32	0.8	9.59		10	1:1
3210	0.85	1.28	0.55	41.1	9.435		10	1.06
3214	1.074	1.578	1.368	18.9	9.52		9.52	1:1
3218	0.935	1.170	0.850	16.8	9.1	R(0.05)	9.1	1:1
3228	1.16	1.26	1.10	6.9	9.52		9.52	1:1
3237	0.9900	0.8950	1.0800	9.4	-----		-----	-----
3246	1.222	1.277	0.733	27.8	9.44		10	1:1
3248	1.029	1.007	1.018	1.1	9.33		9.33	1

normality	suspect		
n	107		
outliers	10		
mean (n)	9.468		
st.dev. (n)	0.1007	RSD=1.1%	
R(calc.)	0.281		

APPENDIX 3

Other reported Analytical Details for sample #19555

lab	ISO/IEC 170251 accredited?	pre-cleaning of test vessel?	cleaning solution	time of cleaning (hours)
110	Yes	No	-----	-----
210	-----	-----	-----	-----
230	Yes	-----	-----	-----
339	Yes	Yes	Nitric acid 20%	One night
348	No	No	-----	-----
362	-----	-----	-----	-----
523	No	Yes	Nitric acid 5%	24
551	Yes	Yes	Nitric acid 20%	4
623	Yes	No	-----	-----
840	Yes	Yes	Nitric acid 5%	24
2102	Yes	Yes	Nitric acid 5%	>4
2115	Yes	No	-----	-----
2117	Yes	No	-----	-----
2129	Yes	No	-----	-----
2132	Yes	No	-----	-----
2135	Yes	No	-----	-----
2137	Yes	Yes	Nitric acid 5%	6
2138	Yes	Other	New/disposable vessel	-----
2139	Yes	Other	New/disposable vessel	-----
2165	Yes	No	-----	-----
2172	Yes	Yes	Nitric acid 4M	4
2184	-----	-----	-----	-----
2201	Yes	Yes	Nitric acid dilute	4
2213	Yes	No	-----	-----
2215	Yes	Yes	Nitric acid 5%	24
2216	Yes	Yes	Nitric acid ~5%	>4
2229	Yes	Yes	-----	-----
2232	Yes	Yes	Nitric acid 5%	>4
2238	Yes	Yes	Nitric acid 10%	24
2241	---	Other	New/disposable vessel	-----
2247	Yes	No	-----	-----
2250	Yes	No	-----	-----
2255	Yes	Yes	Nitric acid 5%	4
2256	Yes	Yes	Nitric acid diluted	>8
2265	No	No	-----	-----
2272	Yes	Other	New/disposable vessel	-----
2289	Yes	No	-----	-----
2290	Yes	---	-----	-----
2293	No	No	-----	-----
2295	Yes	Other	New/disposable vessel	-----
2301	-----	-----	-----	-----
2310	Yes	Yes	Nitric acid diluted & DI water	4
2311	Yes	Yes	Nitric acid 5% & DI water	4
2330	No	Yes	Nitric acid 20%	>6
2347	-----	-----	-----	-----
2350	Yes	No	-----	-----
2352	Yes	Other	New/disposable vessel	-----
2357	Yes	No	-----	-----
2363	Yes	Yes	Nitric acid 5%	4
2365	Yes	Yes	Nitric acid 5%	4
2366	Yes	Yes	Nitric acid 5% & DI water	4
2369	Yes	No	-----	-----
2370	Yes	Yes	Nitric acid 69%	0.33
2374	No	Yes	degreasing solution	-----
2375	Yes	Yes	Nitric acid 5%	4
2377	Yes	No	-----	-----
2378	Yes	Other	New/disposable vessel	-----
2379	No	Yes	Nitric acid 5%	4
2380	Yes	Yes	Nitric acid 5% & DI water	8
2381	Yes	Yes	Nitric acid 5% & DI water	8
2382	-----	-----	-----	-----
2385	Yes	No	-----	-----
2390	Yes	No	-----	-----
2406	Yes	Yes	Nitric acid 10%	>1
2410	Yes	Yes	Nitric acid 4%	6
2415	Yes	No	-----	-----
2429	Yes	Yes	Nitric acid 5%	4
2442	Yes	Yes	Nitric acid 5%	12
2459	Yes	Yes	Nitric acid 5%	4
2462	Yes	Yes	Nitric acid 10%	16
2475	Yes	Yes	Nitric acid 5%	5
2482	Yes	Other	New/disposable vessel	-----
2489	Yes	No	-----	-----
2492	Yes	No	-----	-----

lab	ISO/IEC 170251 accredited?	pre-cleaning of test vessel?	cleaning solution	time of cleaning (hours)
2495	Yes	No	-----	-----
2496	Yes	Yes	Nitric acid 20%	24
2497	Yes	No	-----	-----
2500	Yes	Yes	Nitric acid 2mol/L & DI water	4
2511	-----	-----	-----	-----
2514	Yes	Yes	Nitric acid 5%	4
2522	Yes	No	-----	-----
2532	Yes	Yes	Divers	0.5
2553	Yes	Yes	Aqua Regia	24
2560	Yes	Yes	Nitric acid 10%	24
2563	Yes	Other	Nitric acid 2%	0.08
2582	Yes	Yes	Niric acid 5% and distilled water	Overnight
2590	Yes	No	-----	-----
2624	No	Yes	Nitric acid 5%	20
2629	Yes	Yes	Nitric acid 30%	24
2641	Yes	Yes	-----	6
2652	Yes	Yes	Nitric acid 15% +1 Grade water	-----
2657	Yes	No	-----	-----
2674	Yes	Yes	Nitric acid 5%	4
2703	Yes	No	-----	-----
2705	No	Yes	Nitric acid 5%	1
2719	Yes	Other	-----	-----
2720	Yes	Yes	Nitric acid 5%	4
2741	Yes	Yes	Nitric acid	12
2758	No	No	-----	-----
2804	Yes	Other	New/disposable vessel	-----
2818	Yes	No	-----	-----
2829	Yes	Yes	Nitric acid 5%	4
2832	No	Yes	as required in method	4
2833	-----	-----	-----	-----
2858	Yes	No	-----	-----
2864	Yes	Yes	Nitric acid 2%	24
2878	No	Other	Plastic bag/cleaned with DI water	24
3100	Yes	Yes	Nitric acid 65%	4
3116	Yes	Yes	Nitric acid 5%	4
3118	Yes	No	-----	-----
3146	Yes	Other	New/disposable vessel	-----
3150	Yes	No	-----	-----
3153	Yes	Yes	Nitric acid 5%	4
3154	Yes	No	-----	-----
3160	No	Yes	Soap without metals/distilled water	12
3172	Yes	No	-----	-----
3179	Yes	No	-----	-----
3182	No	Yes	Nitric acid 10%	24
3185	Yes	Yes	Nitric acid 5%	4
3191	Yes	Yes	Nitric acid 5%	8
3197	Yes	Yes	Nitric acid 5%	4
3209	Yes	Yes	DI water	4
3210	Yes	Other	Décon	<1
3214	Yes	Yes	Nitric acid 50%	24
3218	Yes	No	-----	-----
3228	Yes	No	-----	-----
3237	-----	-----	-----	-----
3246	Yes	Yes	Nitric acid 5%	>4
3248	Yes	Yes	Artificial sweat	24

APPENDIX 4

Detailed description on how object was measured for sample #19556

lab	Please, describe as detailed as possible how you have measured and calculated the surface of the object
110	Used a string with lubricant to obtain the length. Took the width to calculate the surface area as a cylinder
210	-----
230	Considered as a cylinder
339	Digital caliper and Excel sheet for calculation
348	Surface of one link in the chain was calculated by approximation to a cylinder (lateral area). Total surface was calculated as six times this area (six links). Dimensional measurements were made with caliper
362	-----
523	Calculated as the area of a cylinder using: π*diameter*length*6
551	Height and diameter of the link were measured. The area was calculated by the cylinder formula
623	Each chain was calculated as a cylinder with top and bottom as a circle. The chain surface area then multiply by 6 as the number of chains
840	-----
2102	The area of 1 ring is calculated by using the formula for a cylinder. This is multiplied by 6 to calculate the area of all 6 rings
2115	We have opened one ring of the chain and we have considered it as a cylinder, then we have multiplied this value for 6 ring
2117	One chain link was opened and stretched to a cylindric shape; the surface of this cylinder was measured; as the chain consists of six chain links this surface area was multiplied by six
2129	One chain link was measured using a digital measuring slide. The result was multiplied by factor 6 (number of chain links)
2132	Find out the circumference and diameter of the link and calculate as area of cylinder
2135	We used wire and digital caliper
2137	5) Total chain Surface Area = 2(¢³d*L) + 2{¢³R*(¢³d)} =2(¢³*1.6*6.7) + 2{¢³*9.4*(¢³*1.6)} = 364.23 mm^2 / 1 chain = 3.6423 cm^2 / 1 chain = 3.6423 cm^2 * 6 chain = 21.8538 cm^2 (unrounded)
2138	We used the vernier calipers for calculated the surface area. = 3.14*(0.175/2)^2*2*6 + 2.3*3.14*0.175*6
2139	Vernier calipers
2165	Use Vernier to calculate manually
2172	For 1 link : measure the length and diameter of 1 link. Side area (Perimeter multiply length) + Top and bottom(two round surface)
2184	-----
2201	Treated the single ring as a cylinder,caculated 6 cylinders's surface area
2213	-----
2215	-----
2216	-----
2229	-----
2232	Assume it is cylinder
2238	Approximate cylinder area, Using vernier caliper measurement
2241	The up/down area is calculated by diameter/length
2247	One link of chain was open and result area as cylinder. This area multiplied by total no of links
2250	chain link formed to a straight shape, measurement by caliper rule, height of chain link = 2,58 cm, diameter of chain link = 0,18cm, calculated as circular cylinder = 1,51 cm² surface area of 1 chain link = 1,51 cm²
2255	Cylindrical Chain . Sued to measuer slide caliper, scale .
2256	1. measure the diameter of a ring (d): 0.176 cm 2. measure the length of a ring loop (L): 2.518 cm 3. Calculate the ring's surface area : 2*pi*(d/2)^2+ pi*d*L 4. times 6 for 6 rings
2265	-----
2272	-Treat ring as cylinder; -Measure the length of cylinder, the diameter of the two head and bottom sections
2289	Straighten one ring, and calculated the surface as a cylinder. And multiply by the number of rings
2290	-----
2293	One link was separated from the chain, then it was opened to its original cylinder form and the area calculated as such. The area was then multiplied times 6 for the complete chain sample
2295	The area was measured by 3D Scanner
2301	Measure Diameter, length and calculate tube surface area
2310	We calculate the surface area of object (circle)using vernier caliper
2311	-----
2330	Digital caliper
2347	-----
2350	It was calculated by the sum of each cylinder area
2352	-----
2357	-----
2363	-----
2365	A=πdl*6
2366	Calculate as cylinder
2369	-----
2370	The six rings are first pulled into a column shape, and after calculating the cylinder, the areas are added
2374	First calculate the surface determination of 1 links chain, and then multiply by 6 to get the total surface
2375	-----
2377	-----
2378	-----
2379	Vernier caliper
2380	Firstly one ring area was calculated among whole chain. One ring like as solid cylinder, so we consider it cylinder law. Then we multiply with six as this chain was contain six chain
2381	Firstly one ring was calculated among whole chain. One ring like as solid cylinder, so we consider it cylinder law. Then we multiply with six as this chain was contain six rings
2382	Measure as hollow column,caculate the surface as follow: =(26.72mm*1.76mm*3.14+3.14/4*1.76mm*1.76mm*2)*6=9.15cm2
2385	Diameter and thickness of the links were measured. From the diameter the length of the sample was calculated. The surface was calculated from diameter and length
2390	Consider only one loop and then straight it. Then consider it as a cylinder and apply formula of surface area of cylinder (i.e πld) then multiply it by total no. of loops. (i.e 6)

lab	Please, describe as detailed as possible how you have measured and calculated the surface of the object
2406	Bend the ring into a straight rod and measure the length of it. Multiple the length of one ring with no. of ring (6pcs). The length of one ring is 2.585cm and the radius of rod is 0.179/2 cm
2410	
2415	Total area of the chain is 6 times of area of 1 piece Consider area of 1 piece of the chain as area of 2 rods and a ring
2429	The area of the sample is regarded as a regular and easily calculated area by filling
2442	Separate the each part of chain and measure each surface are with digital caliper
2459	Area = 2πrh + 2πr2
2462	Use ruler and vernier caliper
2475	We have use the formula below to calculate the Side area of a cylinder: area=πdh with d= 0.18cm and h= 3cm
2482	Digital caliper
2489	Measured with Vernier caliber, Length, width and thickness and calculated. Holes portions substracted
2492	Each chain is separated into 2 half-circle and 2 straight line
2495	Calculation based on single ring: lenght of the ring measured with a wire; surface calculated as cylinder. Value multiplied by 6 (the number of rings)
2496	Straighten the sample, then measure and calculate with vernier caliper
2497	3D scanner
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
2511	-----
2514	-----
2522	Our lab used the digital vernier caliper
2532	By digital vernier caliber
2553	Draw the sample, divide to desired shapes and calculate the total surface
2560	The sample consists 6 twisted ring, considered each as a whole cylinder. We used formula for Surface Area Measurement is A=(2πr2+2πrh)x6
2563	One Piece abstracted to a cylindrical shape and Determination of the sheath surface; multipied for 6
2582	Measure the surface area of one loop and multiplied it by number of loops in the chain
2590	I used the following geometrical figures: cylinder and bull
2624	Measured with a digital caliper, approximating the links to elliptical shapes
2629	Spread each unit of the chain to "cylinder" then calculated around surface area
2641	Caculated each ring's surface area,then sum together
2652	-----
2657	Using caliper
2674	Use vernier to calculate manually
2703	Link opened and straightened. Surface area of cylinder formula used. Height measured with calibrated ruler (26mm). Diameter measured with calibrated calipers (1.79mm), radius 1/2 of this (0.895mm). Surface area for 1 link calculated as 151.242 sqmm. Multiplied by 6 for whole chain
2705	6* Elliptic Torus
2719	-----
2720	Cut the metal circle, test and calculate the area
2741	The chain have 06 links. One link should be lengthened; it is a cylinder
2758	-----
2804	Treat the ring as a straight metal round rod and calculate the surface area (2*pi*r*h) accordingly
2818	S=(πdl+πr2*2)*6
2829	the area of a ring was calculated through the area of a cylinder
2832	We have cut one part of the chain, stratched it and finally measured the dimensions (lenght and thickness)with calibre
2833	Remove one link and Count as solid cylinder after that multiply with 6 .
2858	Straighten the sample and measure the length and diameter
2864	Measure 1 unit and then multiple 6 for the full sample
2878	Measure the diameter and total length of the chain, approximate to the cylinder area formula
3100	By calculating the circumference and the length of each link to obtain the surface area of each link and finally summed together
3116	One sample was cut and measured in diameter and length. Sample are measured in 4 point using calibrated Vernier Caliper. The area calculated using the formula of the "tube blanket" area. The final result of the area multiplied by 6 rings
3118	One chain link bent up, calculated as cylinder multiplied by 6 links
3146	-----
3150	Geometric approximation
3153	-----
3154	One link was opened to measure like a cylinder. Then, this surface was multiplied by six
3160	-----
3172	One chain link was opened and stretched in a cylindric shape; the surface of this cylinder was measured; as the chain consists of six chain links this surface area was multiplied by six
3179	By using equation(2 x 3.14 x rh)x6 When r is radius of chain, h is length of chain and multiply with 6 chain
3182	Straighten one repeating unit of the chain sample and calculate it's surface area as a cylinder ,then multiply the number of repeating units:6
3185	The sample is divided into several small cylinders, which can be see in the remarks
3191	Digital caliper was used to determine the surface area. Only the are of 1 ring of the chain was measured, and the area was multiplied by 6 then. The area of the ring is calculated such that it is opened and thought as a cylinder
3197	First calculate the area of a ring. Then multiply the area of this ring by 6 to get the area of the chain
3209	6x(Tore: 4x3.14x3.14xRxr + 2cylindres:2x 2x3.14xrxh)
3210	Measure the length and width of a single metal ring
3214	r=0.0885cm,L=2.6cm;S=2πrL*6
3218	-----
3228	-----
3237	-----
3246	Using a thin string to determine the circumference of the ring
3248	Used a string with lubricant to obtain the length. Took the width to calculate the surface area as a cylinder

APPENDIX 5

Number of participants per country

7 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	BULGARIA
1 lab in	CAMBODIA
4 labs in	FRANCE
12 labs in	GERMANY
1 lab in	GUATEMALA
11 labs in	HONG KONG
6 labs in	INDIA
3 labs in	INDONESIA
8 labs in	ITALY
1 lab in	LUXEMBOURG
1 lab in	MAURITIUS
1 lab in	MEXICO
1 lab in	MOROCCO
36 labs in	P.R. of CHINA
2 labs in	PAKISTAN
1 lab in	SINGAPORE
5 labs in	SOUTH KOREA
2 labs in	SPAIN
2 labs in	SRI LANKA
1 lab in	SWITZERLAND
3 labs in	TAIWAN R.O.C.
2 labs in	THAILAND
1 lab in	THE NETHERLANDS
1 lab in	TUNISIA
4 labs in	TURKEY
2 labs in	U.S.A.
1 lab in	UNITED KINGDOM
7 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 = possibly an error in calculations
U = test result possibly reported in a different unit
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

Literature:

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