Results of Proficiency Test
Nickel Release and Surface determination
June 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

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 2019/2020 it was decided to continue the proficiency test for the analysis of Nickel release and surface determination.
In this interlaboratory study 108 laboratories in 26 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 #20620 positive on Nickel release and one piece of a metallic cufflink labelled #20621 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 not plated or coated. The dimensions of each item were approximately 1.2x1.2x0.2cm and the hole had a diameter of approximately 0.4cm. Three items were packed in a small plastic bag and vacuum sealed to avoid scratching of the items. Each bag was labelled #20620. The homogeneity of the subsamples was checked by determination of Nickel release using test method EN1811:11+A1:15 on six stratified randomly selected subsamples. Please note, a subsample is one bag with three items.

	Nickel Release in µg/cm^2/week
sample #20620-1	2.7
sample #20620-2	2.5
sample #20620-3	2.5
sample #20620-4	2.5
sample #20620-5	2.7
sample #20620-6	2.5

Table 1: homogeneity test results of subsamples #20620

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

	Nickel Release in µg/cm^2/week
r (observed)	0.3
reference method	Horwitz *)
0.3 x R (reference method)	0.4

Table 2: evaluation of the repeatability of subsamples #20620

*) The Horwitz formula is converted to µg/cm^2/week unit

The calculated repeatability was in agreement with 0.3 times the target reproducibility estimated from the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

Surface Determination
A batch of metal cufflinks was obtained from a local supplier. From this batch 132 small plastic bags were filled with one cufflink each and labelled #20261. 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 #20620 and one sample #20261 were sent on May 20, 2020.

2.5 ANALYZES

The participants were requested to determine Nickel Release on sample #20620 and to determine surface only on sample #20621, 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.

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.

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 was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ISO reproducibilities, 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. 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 should be done in order to evaluate whether the reported test results are fit-for-purpose.

The z-scores were calculated in accordance with:

$z_{(target)}$ = (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

During the execution of this proficiency test no severe problems were encountered due to the Covid-19 pandemic. Three participants reported test results after the reporting deadline and four other participants did not report any test results at all. In total 104 participants reported 205 test results for Nickel Release and Surface Determination. Observed were 11 outlying test results, which is 5.4%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

The data sets proved to have a normal Gaussian distribution.

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 and ranges from 54% at 0.3 µg Ni/cm^2/week up to 32% at 10 µg Ni/cm^2/week.

Sample #20620

Nickel Release: This determination may be problematic. Seven statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the target reproducibility estimated from the Horwitz equation.

Sample #20621

Surface Determination: This determination on the Cufflink may be problematic. Four statistical outliers were observed in the reported range of 3.62-9.49cm^2. No official test method exists for Surface Determination. Therefore, no z-scores were calculated. The variation for this sample of 3.5% is better than the observed variation in previous PT’s in which the Surface Determination was evaluated (4.9-13%) but is larger compared to the variation of the Surface Determination of the much simpler shaped sample #20620 (2.5%).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test 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 derived from the estimated target reproducibility are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(target)
Nickel Release	µg/cm^2/week	95	1.26	1.02	0.63
Surface	cm^2	100	3.90	0.27	n.a.

Table 3: reproducibilities of test results on sample #20620

It can be concluded, without further statistical calculations, that the group of participating laboratories had problems with the analysis of Nickel Release when compared to the Horwitz target reproducibility.

Parameter	unit	n	average	2.8 * sd	R(target)
Surface Determination	cm^2	98	8.26	0.81	n.a.

Table 4: reproducibility of test results on sample #20621

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2020 WITH PREVIOUS PTS

	June 2020	June 2019 *)	May 2018 *)	May 2017 *)	May 2016 *)
Number of reporting laboratories	104	127	113	122	125
Number of test results	205	126	112	122	124
Number of statistical outliers	11	5	4	14	8
Percentage statistical outliers	5.4%	4.0%	3.6%	11%	6.5%

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 2020	June 2019	May 2018	May 2017	2016-2014
Nickel Release	29%	30%	44%	26%	18 - 31%
Surface Determination	2.5 – 3.5%	1.1 - 7%	1.3 - 13%	1.3 - 6.7%	1.7 - 10%

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

The uncertainty of the Nickel Release determination is in line with the average uncertainty from the previous years.

Remarkedly, the uncertainty of the Surface Determination (2.5%) for sample #20260 is higher with the uncertainty of previous samples, while for sample #20261 it has been improved (3.5%).

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For the Nickel Release sample #20620 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, 2% of the reporting participants have not done any pre-treatment and 8% did not answer this question, see appendix 3.
- About 75% 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 between 1.5 and 4 times as much volume of test solution than sample area.
- In total 103 laboratories reported the average surface area, see appendix 2. The average surface area varied from 3.6 to 9.5cm^2 with a mean of 3.9 cm^2.

For the Surface Determination sample #20621 only one question was requested: a detailed description on how the surface area was measured and calculated which was answered by 66% 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 huge; from 0.4 up to 85.2 RSD(%). Only 34 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). Remarkedly, when evaluating the test results of only those 34 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.

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 3.5 and 4.5mL. 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 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.

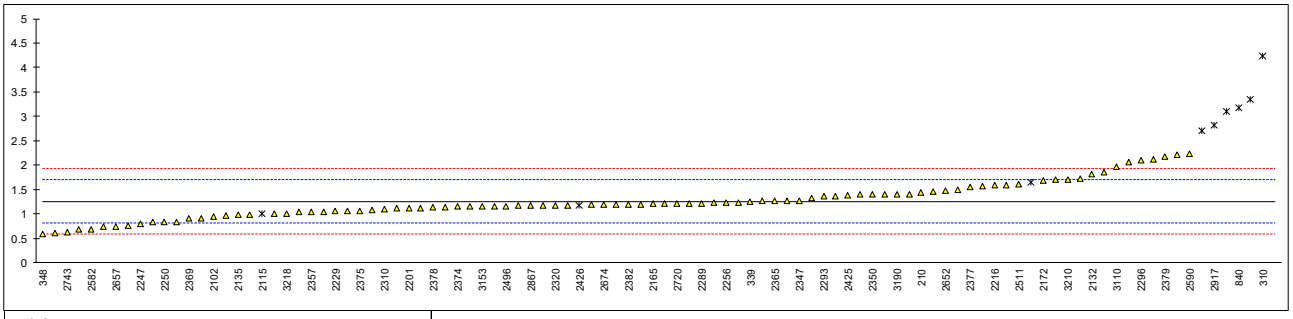
APPENDIX 1

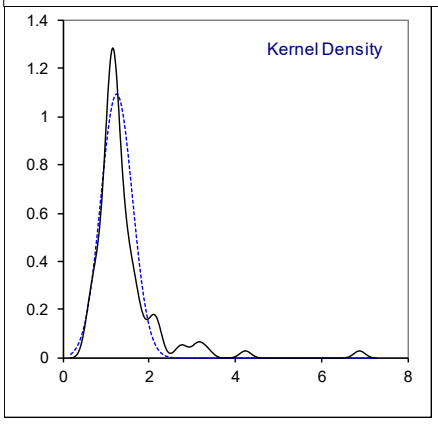
Determination of Nickel Release on sample #20620; average result of three replicates in µg/cm²/week

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	EN1811	1.43		0.76	
230		-----		-----	
310	EN1811	4.234	C,R(0.01)	13.26	First reported 3.87
339	In house	1.243		-0.08	
348	EN1811 + A1	0.592		-2.98	
551	EN1811 + A1	1.4080		0.66	
623	EN1811 + A1	1.566		1.36	
840	EN1811	3.172	R(0.01)	8.52	
841	EN1811 + A1	6.87	R(0.01)	25.00	
2102	EN1811 + A1	0.94483		-1.40	
2115	EN1811	1.0		-1.16	
2120	EN1811 + A1	2.7	R(0.05)	6.42	
2129	EN1811	0.828		-1.93	
2132	EN1811 + A1	1.8224		2.51	
2135	EN1811	0.9877		-1.21	
2165	EN1811 + A1	1.21		-0.22	
2172	EN1811 + A1	1.681		1.88	
2184	EN1811 + A1	1.078		-0.81	
2201	EN1811 + A1	1.118		-0.63	
2216	EN1811	1.5852		1.45	
2229	EN1811 + A1	1.058		-0.90	
2238	EN1811 + A1	1.180		-0.36	
2241	EN1811 + A1	1.861		2.68	
2247	EN1811	0.802		-2.04	
2250	EN1811 + A1	0.8300		-1.92	
2256	EN1811 + A1	1.240		-0.09	
2266	EN1811 + A1	1.655		1.76	
2284	EN1811 + A1	1.594		1.49	
2289	EN1811 + A1	1.222		-0.17	
2290	EN1811	1.203	C	-0.25	First reported 2.271
2293	EN1811 + A1	1.369		0.49	
2295	EN1811	1.5		1.07	
2296	EN1811 + A1	2.105055059		3.77	
2310	EN1811 + AC	1.09		-0.76	
2311	EN1811 + A1	1.036		-1.00	
2320	EN1811	1.178		-0.37	
2347	EN1811 + A1	1.27		0.04	
2350	EN1811 + A1	1.402		0.63	
2352	EN1811	1.192		-0.30	
2357	EN1811	1.039		-0.98	
2363	EN1811 + A1	1.11		-0.67	
2365	EN1811 + A1	1.2667		0.03	
2366	EN1811 + A1	1.152		-0.48	
2369	EN1811 + A1	0.91		-1.56	
2370	EN1811	1.24		-0.09	
2374	EN1811 + AC	1.15		-0.49	
2375	EN1811 + A1	1.07		-0.85	
2377	EN1811 + A1	1.55		1.29	
2378	EN1811	1.13		-0.58	
2379	EN1811 + A1	2.167		4.04	
2380	EN1811 + A1	1.403		0.64	
2381	EN1811	1.370		0.49	
2382	EN1811	1.20		-0.27	
2385		-----		-----	iis calc average 1.883: z(targ) = 2.78
2390	EN1811 + A1	0.74		-2.32	
2406	EN1811 + A1	0.9146		-1.54	
2410		2.22		4.28	
2425	EN1811 + A1	1.39		0.58	
2426	EN1811 + A1	1.18		-0.36	
2429	EN1811 + AC	1.23		-0.13	
2442	EN1811 + A1	1.172		-0.39	
2452	EN1811 + A1	0.754		-2.26	
2475	EN1811	1.398		0.62	
2489	EN1811	1.0		-1.16	
2496	EN1811 + A1	1.158		-0.45	
2508	EN1811	2.12		3.83	
2511	EN1811	1.61		1.56	
2532	EN1811	1.04		-0.98	
2538		-----		-----	iis calc average 0.835: z(targ) = -1.89
2549	EN1811 + A1	1.22		-0.18	
2560	EN1811	1.46		0.89	
2563		-----		-----	
2582	EN1811 + A1	0.688		-2.55	

lab	method	value	mark	z(targ)	remarks
2590	EN1811	2.2286	C	4.32	First reported 2.2967
2591	EN1811	1.2126		-0.21	
2624	EN1811 + A1	0.99		-1.20	
2643	EN1811 + A1	3.10	R(0.01)	8.20	
2652	EN1811	1.4684		0.93	
2657	EN1811 + A1	0.74359		-2.30	
2668	EN1811	1.15		-0.49	
2674	EN1811	1.193		-0.30	
2713		-----		-----	
2720	EN1811 + A1	1.214		-0.20	
2743	EN1811 + A1	0.62200211		-2.84	
2749	EN1811	0.832	C	-1.91	First reported 0.315
2804	EN1811	1.193		-0.30	
2818	EN1811 + A1	1.120		-0.62	
2864	EN1811 + A1	1.32	C	0.27	First reported 2.21
2867	EN1811	1.172		-0.39	
2900	EN1811	3.3549	R(0.01)	9.34	
2917	EN1811 + A1	2.82	R(0.05)	6.95	
3100	EN1811 + A1	1.1349		-0.56	
3110	EN1811	1.97		3.16	
3116	EN1811 + A1	1.2696		0.04	
3118	EN1811 + A1	0.6819		-2.58	
3153	EN1811 + A1	1.151		-0.49	
3172	EN1811 + A1	1.72		2.05	
3176	EN1811 + A1	0.616		-2.87	
3182	EN1811	2.068		3.60	
3185	EN1811 + A1	1.175		-0.38	
3190	EN1811	1.407		0.66	
3197	EN1811 + A1	1.06		-0.89	
3210	EN1811	1.71		2.01	
3218	EN1811	1.01		-1.11	
3225	EN1811	1.70		1.96	
3228	EN1811 + A1	1.263		0.01	
3237	EN1811 + A1	0.97		-1.29	

				Only RSD <4% between		Only with ratio 1:1	
	normality	OK		not OK		OK	
	n	95		34		71	
	outliers	7		0		6	
	mean (n)	1.260		1.235		1.256	
	st.dev. (n)	0.3655	RSD = 29%	0.3141	RSD = 25%	0.3524	RSD = 28%
	R(calc.)	1.023		0.879		0.987	
	st.dev.(Horwitz)	0.2244		0.2206		0.2238	
	R(Horwitz)	0.628		0.618		0.627	
Compare							
	R(EN1811:11+A1:15)	0.419					



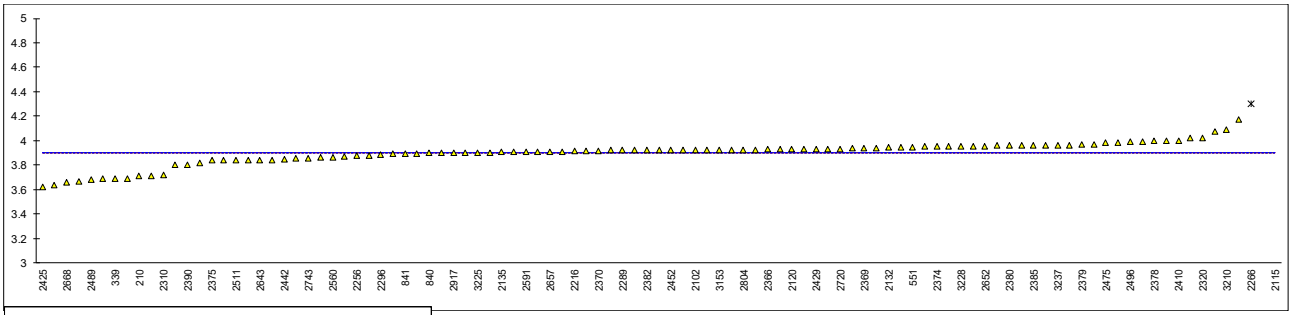


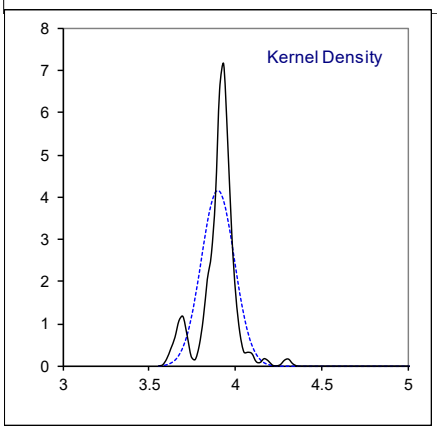
Determination of Surface on sample #20621; results in cm^2

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
210	see appendix 4	7.938		-----	
230		-----		-----	
310	see appendix 4	7.62		-----	
339	see appendix 4	8.82		-----	
348	see appendix 4	8.485		-----	
551	see appendix 4	5.20777	C,R(0.01)	-----	First reported 9.5897
623	see appendix 4	8.078		-----	
840	see appendix 4	8.502		-----	
841	see appendix 4	8.51		-----	
2102	see appendix 4	8.9469		-----	
2115	see appendix 4	8.309		-----	
2120	see appendix 4	8.21		-----	
2129	see appendix 4	9.16		-----	
2132		-----		-----	
2135	see appendix 4	8.633		-----	
2165	see appendix 4	8.61		-----	
2172	see appendix 4	8.274		-----	
2184	see appendix 4	8.414		-----	
2201	see appendix 4	8.195		-----	
2216	see appendix 4	8.34		-----	
2229	see appendix 4	8.602		-----	
2238	see appendix 4	8.149		-----	
2241	see appendix 4	8.171		-----	
2247	see appendix 4	8.44		-----	
2250	see appendix 4	7.771		-----	
2256	see appendix 4	8.482		-----	
2266	see appendix 4	7.8		-----	
2284	see appendix 4	8.313		-----	
2289	see appendix 4	8.13		-----	
2290	see appendix 4	8.00		-----	
2293	see appendix 4	9.951	R(0.01)	-----	
2295	see appendix 4	8.3		-----	
2296	see appendix 4	8.442		-----	
2310	see appendix 4	8.1		-----	
2311	see appendix 4	8.14		-----	
2320	see appendix 4	8.439		-----	
2347	see appendix 4	8.30		-----	
2350	see appendix 4	8.021		-----	
2352	see appendix 4	8.09		-----	
2357	see appendix 4	8.218		-----	
2363	see appendix 4	8.29		-----	
2365	see appendix 4	8.259		-----	
2366	see appendix 4	8.28		-----	
2369	see appendix 4	8.17		-----	
2370	see appendix 4	8.273		-----	
2374	see appendix 4	8.25		-----	
2375	see appendix 4	8.1		-----	
2377	see appendix 4	8.27		-----	
2378	see appendix 4	8.05		-----	
2379	see appendix 4	7.983		-----	
2380	see appendix 4	8.512		-----	
2381	see appendix 4	8.483		-----	
2382	see appendix 4	8.17		-----	
2385	see appendix 4	8.58		-----	
2390	see appendix 4	7.94		-----	
2406	see appendix 4	8.29		-----	
2410	see appendix 4	8.13		-----	
2425	see appendix 4	8.42		-----	
2426	see appendix 4	23.1	R(0.01)	-----	
2429	see appendix 4	8.07		-----	
2442	see appendix 4	8.3402		-----	
2452	see appendix 4	8.344		-----	
2475	see appendix 4	8.26		-----	
2489	see appendix 4	8.75		-----	
2496	see appendix 4	8.51		-----	
2508	see appendix 4	8.84		-----	
2511	see appendix 4	8.251		-----	
2532	see appendix 4	8.6		-----	
2538	see appendix 4	8.286		-----	
2549	see appendix 4	8.28		-----	
2560	see appendix 4	8.125		-----	
2563		-----		-----	
2582	see appendix 4	7.780		-----	

lab	method	value	mark	z(targ)	remarks
2590	see appendix 4	7.6216		-----	
2591	see appendix 4	8.55		-----	
2624	see appendix 4	8.82	C	-----	First reported 11.66
2643	see appendix 4	6.98	R(0.01)	-----	
2652	see appendix 4	8.2109		-----	
2657	see appendix 4	8.705		-----	
2668	see appendix 4	8.24		-----	
2674	see appendix 4	8.324		-----	
2713		-----		-----	
2720	see appendix 4	8.173		-----	
2743	see appendix 4	7.569978	C	-----	First reported 7.01860713
2749	see appendix 4	7.776		-----	
2804	see appendix 4	8.120		-----	
2818	see appendix 4	8.021		-----	
2864	see appendix 4	8.15		-----	
2867	see appendix 4	8.274		-----	
2900		-----		-----	
2917	see appendix 4	7.64		-----	
3100	see appendix 4	8.18		-----	
3110	see appendix 4	8.18		-----	
3116	see appendix 4	8.57		-----	
3118	see appendix 4	7.896	C	-----	First reported 7.3365
3153	see appendix 4	8.4998		-----	
3172	see appendix 4	8.252		-----	
3176	see appendix 4	8.34		-----	
3182	see appendix 4	7.832		-----	
3185	see appendix 4	8.278		-----	
3190	see appendix 4	8.15		-----	
3197	see appendix 4	8.31		-----	
3210	see appendix 4	7.90		-----	
3218	see appendix 4	8.18		-----	
3225	see appendix 4	8.39		-----	
3228	see appendix 4	8.071		-----	
3237	see appendix 4	8.08		-----	

normality	OK	
n	98	
outliers	4	
mean (n)	8.2566	
st.dev. (n)	0.28848	RSD = 3.5%
R(calc.)	0.8077	
st.dev.(lit)	n.a.	
R(lit)	n.a.	





APPENDIX 2

Determination of Nickel Release and some Analytical Details on subsamples #20620

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	-----		-----		-----		-----	-----		---	---
210	1.66		1.53		1.09		20.9	3.708		---	---
230	-----		-----		-----		-----	-----		---	---
310	4.670	C	3.491	C	4.542	C	15.3	3.92		4	1:1
339	1.04		1.29		1.40		14.8	3.69		---	---
348	0.633		0.575		0.568		6.0	3.91		4	1:1
551	1.1408		1.5865		1.4966		16.7	3.9489		3.94	1:1
623	1.285		1.454		1.955		22.3	3.67		3.66	1:1
840	3.682		2.663		4.370		24.0	3.900		4	1.03:1
841	6.20		6.22		8.19		16.6	3.89		4	1:1
2102	0.98245		0.90653		0.94550		4.0	3.9209		4	1:1
2115	0.96		0.92		1.04		6.3	9.49	R(0.01)	9.5	1:1
2120	1.39	C	1.543		0.933		24.6	3.93		4	1:1
2129	0.700		0.865		0.918		13.7	3.87		4	1:1
2132	1.6382		2.0178		1.819		10.4	3.943		3.95	1:0.998
2135	1.036		0.921		1.006		6.0	3.906		10	2.5:1
2165	1.21		1.23		1.19		1.7	3.96		4	1.01:1
2172	1.780		1.494		1.768		9.6	3.982		6	6:3.98
2184	1.070		1.080		1.083		0.6	3.861		4	1:1
2201	1.328		0.9819		1.044		16.5	3.915		3.9	1:1
2216	1.0318		2.5141		1.2096		51.1	3.9127		3	1:1
2229	1.082		1.011		1.082		3.9	3.95		3.95	1:1
2238	1.522		0.882		1.137		27.3	3.93		3.9	1:1
2241	1.838		1.889		1.857		1.4	3.92		4 (25)	3.92:4
2247	0.729		0.830		0.847		8.0	4.023		4	1:1
2250	0.6116		0.9023		0.9761		23.2	3.890		3.89	1:1
2256	1.1630		1.307		1.251		5.9	3.878		4	1.03:1
2266	1.584		1.751		1.630		5.2	4.3	R(0.01)	4.3	1:1
2284	1.291		1.847		1.644		17.7	3.923		5	1.28:1
2289	1.129		1.252		1.285		6.7	3.92		3.9	1:1
2290	2.265		2.353		2.196		3.5	3.93	C	2.2	1:1
2293	1.373		1.441		1.294		5.4	3.963		10	2.5:1
2295	2.1		1.7		0.6		53.0	3.8		5	5:3.8
2296	2.287916347		2.197657395		1.829591435		11.5	3.883		4	1.03:1
2310	1.1		0.98		1.2		10.1	3.72	C	5	1:1
2311	1.10		1.01		1.00		5.3	3.97		4	1:1
2320	1.172		1.180		1.182		0.4	4.024		5	1.24:1
2347	0.87		1.38		1.56		28.2	3.90		---	---
2350	1.451		1.432		1.324		4.9	4.172		5	1:1
2352	1.230		1.208		1.138		4.0	4.00		4	4:4
2357	0.532		1.040		1.544		48.7	3.951		---	---
2363	1.04		1.11		1.18		6.3	3.95		3.95	1:1
2365	1.2726		1.2321		1.2953		2.5	3.948		3.95	1:1
2366	1.186		1.123		1.146		2.8	3.93		4	1.02:1
2369	0.902		0.911		0.910		0.5	3.94		---	---
2370	1.25		1.24		1.22		1.2	3.918		4	1:1
2374	1.10		1.15		1.20		4.3	3.95		3.95	1:1
2375	1.10		1.10		1.02		4.3	3.84		3.84	1:1
2377	1.37		1.50		1.77		13.2	3.96		4	1:1
2378	1.15		1.16		1.08		3.9	4		4	1:1
2379	2.253		2.123		2.125		3.4	3.970		4	1:1
2380	1.408		1.384		1.417		1.2	3.960		6	1:1
2381	1.394		1.353		1.363		1.6	3.819		4	1:1
2382	1.11		1.29		1.20		7.5	3.92		10	1:1
2385	2.216		1.462		1.970		20.4	3.96		6	---
2390	0.71		0.72		0.79		5.9	3.80		3.8	1:1
2406	1.0020		0.7991		0.9426		11.4	3.92		4	1:1
2410	2.36		2.20		2.09		6.1	4.0	C	5	2:1
2425	1.44		1.35		1.37		3.4	3.62		4	1:1
2426	1.17		1.18		1.18		0.5	6.45	R(0.01)	6.5	1:1
2429	1.28		1.10		1.32		9.5	3.93		10	2.54 : 1
2442	1.160		1.168		1.188		1.2	3.851		4	1:1
2452	0.781		0.793		0.688		7.6	3.92		3.92	1:1
2475	2.766		0.584		0.845		85.2	3.98		3.98	1:1
2489	0.99		1.04		0.97		3.6	3.68		3.68	1:1
2496	1.202		1.418		0.855		24.5	3.99		---	---
2508	2.16		2.28		1.92		8.6	3.84		15	3.9:1
2511	1.62		1.48		1.73		7.8	3.84		---	---
2532	1.06		1.20		0.86		16.4	3.71		3.7	1:1
2538	0.903		0.749		0.853		9.4	3.855		10 - 20	1:1
2549	1.51		1.15		1		21.5	3.84		4	1.04:1
2560	1.44		1.49		1.44		2.0	3.861		50	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)
2563	-----	-----	-----	-----	-----		---	---
2582	0.711	0.694	0.659	3.9	3.9926		3	1:1
2590	2.1135 C	2.2583	2.3139	4.6	3.6394		5	1:1
2591	1.1981	1.2328	1.2069	1.5	3.91		4	1:1
2624	1.03	0.96	0.98	3.6	3.91		4	1:1
2643	2.82	3.13	3.34	8.4	3.84		4	1:1
2652	1.4825	1.4519	1.4709	1.1	3.9516		---	---
2657	1.05128	0.53846	0.64103	36.5	3.910		4	1:1
2668	1.28	0.97	1.27	15.0	3.66		5	1.36:1
2674	1.171	1.194	1.215	1.8	3.921		5	1.28:1
2713	-----	-----	-----	-----	-----		---	---
2720	1.191	1.252	1.201	2.7	3.933		3.93	1:1
2743	0.88296993	0.49722147	0.48581493	36.3	3.857436		10	1:2.5
2749	0.310	0.298	0.337	6.3	3.69		6	1.6:1
2804	1.246	1.209	1.124	5.2	3.925		3.9	1:1
2818	0.919	1.208	1.232	15.6	3.904		3.9	1:1
2864	2.28	2.12	2.23	3.7	3.96		4	1:1
2867	1.191	1.104	1.221	5.2	3.893		3.9	1:1
2900	3.7286	3.1328	3.2033	9.7	4.0771	C	5	1:1
2917	3.32	2.159	3.013	21.3	3.90		10	2.5:1
3100	1.0992	1.2443	1.0611	8.5	3.93		3.93	1:1
3110	1.8	2.0	2.1	7.8	3.94	C	4	1:1
3116	1.3286	1.2725	1.2077	4.8	3.90		4	1:1
3118	0.7660	0.6276	0.6520	10.8	3.9350		4	1:1
3153	1.055	1.077	1.321	12.8	3.922		3.9	1:1
3172	-----	-----	-----	-----	3.687		3.7	1:1
3176	0.608	0.623	0.616	1.2	3.84		6	1.5:1
3182	2.060	2.106	2.039	1.7	3.926		3.93	1:1
3185	1.195	1.175	1.154	1.7	-----		4	4:3.95
3190	1.955	1.673	0.593	51.1	3.92		3.92	1:1
3197	1.29	0.86	1.03	20.4	3.88		3.88	1:1
3210	1.62	1.90	1.62	9.4	4.0911		10	2.44:1
3218	1.00	1.07	0.95	6.0	3.91		3.91	1:1
3225	1.86	1.61	1.63	8.2	3.9		3.9	1:1
3228	1.237	1.291	1.262	2.1	3.95		3.95	1:1
3237	0.80	1.14	0.97	17.5	3.96		5	1:1
				normality	suspect			
				N	100			
				outliers	3			
				mean (n)	3.899			
				st.dev. (n)	0.0959	RSD = 2.5%		
				R(calc)	0.269			

Lab 310: First reported 4.268, 3.191, 4.151
Lab 2120: First reported 5.56
Lab 2290: First reported 2.2
Lab 2310: First reported 3.46
Lab 2410: First reported 2.27
Lab 2590: First reported 2.3248
Lab 2900: First reported 7.0771
Lab 3110: First reported 8.65

APPENDIX 3

Other reported Analytical Details for sample #20620

lab	ISO/IEC 170251 accredited?	pre-cleaning of test vessel?	cleaning solution
110	---	---	
210	No	---	
230	---	---	
310	No	No, new/disposable test vessel(s) were used	
339	No	---	
348	Yes	No, new/disposable test vessel(s) were used	
551	Yes	No, new/disposable test vessel(s) were used	
623	Yes	No, new/disposable test vessel(s) were used	
840	Yes	No, new/disposable test vessel(s) were used	
841	Yes	No, new/disposable test vessel(s) were used	
2102	Yes	Yes, the previously used test vessel(s) were pre-treated	A vessel shall be pre-treated by being stored in a solution of dilute nitric acid 5% v/v for at least 4 hours. After acid treatment, the vessel is rinsed with deionized water and dried.
2115	Yes	No, the previously used test vessel(s) were not pre-treated	
2120	No	Yes, the previously used test vessel(s) were pre-treated	
2129	Yes	No, new/disposable test vessel(s) were used	
2132	Yes	No, new/disposable test vessel(s) were used	
2135	Yes	Yes, the previously used test vessel(s) were pre-treated	in 5% HNO3 acid overnight
2165	Yes	No, new/disposable test vessel(s) were used	
2172	Yes	Yes, the previously used test vessel(s) were pre-treated	Immerse test vessel in 4M nitric acid for 4 hours.
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
2216	Yes	Yes, the previously used test vessel(s) were pre-treated	5% nitric acid soak
2229	Yes	Yes, the previously used test vessel(s) were pre-treated	Degrease the sample before test.
2238	Yes	Yes, the previously used test vessel(s) were pre-treated	Dip in 5% nitric acid for 24 hours
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	
2256	Yes	Yes, the previously used test vessel(s) were pre-treated	Soak the vessel in 4M nitric acid for at least 4 hours Rinse with tap water then DI water Completely dried before use
2266	Yes	No, the previously used test vessel(s) were not pre-treated	
2284	Yes	No, the previously used test vessel(s) were not pre-treated	Stored the vessels and holders in 5% HNO3 for 12h, and rinsed them with DI water and let them dry.
2289	Yes	No, new/disposable test vessel(s) were used	
2290	Yes	Yes, the previously used test vessel(s) were pre-treated	soak overnight with HNO3 10%, rinse thoroughly with ultrapure water
2293	No	No, new/disposable test vessel(s) were used	
2295	Yes	No, new/disposable test vessel(s) were used	
2296	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.
2320	Yes	Yes, the previously used test vessel(s) were pre-treated	Pretreated by stored in 5% nitric acid for 4 hours
2347	---	---	
2350	Yes	No, new/disposable test vessel(s) were used	
2352	Yes	No, new/disposable test vessel(s) were used	
2357	Yes	---	
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	No, new/disposable test vessel(s) were used	
2369	---	---	
2370	Yes	Other option, please describe in remarks below	new/disposable test vessel (s) were used¡Awith solution: 10%.HNO3 for 20mins.
2374	No	Yes, the previously used test vessel(s) were pre-treated	Gently swirl the sample for (2) min in degreasing solution by a mechanical shaker.
2375	Yes	No, new/disposable test vessel(s) were used	
2377	Yes	No, new/disposable test vessel(s) were used	
2378	Yes	No, the previously used test vessel(s) were not pre-treated	
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	At first submerge the apparatus in 5% HNO3 solution. Then after 24 hours wash these apparatuses by detergent & then wash by distilled water.
2381	Yes	Yes, the previously used test vessel(s) were pre-treated	All test vessels are pretreated by 5% HNO3 & then rinse the vessels by Deionized water.
2382	Yes	Yes, the previously used test vessel(s) were pre-treated	5%HNO3 soaking for 4h, wash with deionized water and dry
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	

lab	ISO/IEC 170251 accredited?	pre-cleaning of test vessel?	cleaning solution
2410	Yes	Yes, the previously used test vessel(s) were pre-treated	Fill the vessel with 5% HNO3 and leave the vessel over 4 hours.
2425	Yes	No, new/disposable test vessel(s) were used	
2426	Yes	No, new/disposable test vessel(s) were used	
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	
2452	No	Yes, the previously used test vessel(s) were pre-treated	4 hours in HNO3 10%(V/V)
2475	Yes	Yes, the previously used test vessel(s) were pre-treated	HNO3 5%
2489	Yes	No, new/disposable test vessel(s) were used	
2496	Yes	Yes, the previously used test vessel(s) were pre-treated	
2508	Yes	No, new/disposable test vessel(s) were used	
2511	---	---	
2532	Yes	No, new/disposable test vessel(s) were used	
2538	Yes	Vessels weren't pre-treated	
2549	Yes	No, new/disposable test vessel(s) were used	Not Applicable
2560	---	---	Test vessel has been cleaned with 10 % nitic acid for 12 hours.
2563	---	---	
2582	Yes	No, new/disposable test vessel(s) were used	
2590	Yes	No, new/disposable test vessel(s) were used	
2591	Yes	No, the previously used test vessel(s) were not pre-treated	No pre-treatment performed
2624	No	No, new/disposable test vessel(s) were used	
2643	Yes	No, new/disposable test vessel(s) were used	
2652	Yes	---	
2657	Yes	No, new/disposable test vessel(s) were used	
2668	Yes	No, new/disposable test vessel(s) were used	5 % HNo3 with 4 hrs
2674	Yes	Yes, the previously used test vessel(s) were pre-treated	5%HNO3 steep 4 hours
2713	---	---	
2720	Yes	Yes, the previously used test vessel(s) were pre-treated	Soak in 5% nitric acid for 4 hours
2743	Yes	Yes, the previously used test vessel(s) were pre-treated	Wash with HNO3diluited solution in ultrasonic bath (10 minutes) Wash with H2O milliQ. Dry in oven (100°C)
2749	Yes	No, new/disposable test vessel(s) were used	
2804	Yes	No, new/disposable test vessel(s) were used	
2818	Yes	No, new/disposable test vessel(s) were used	
2864	Yes	No, new/disposable test vessel(s) were used	
2867	Yes	No, new/disposable test vessel(s) were used	
2900	Yes	Yes, the previously used test vessel(s) were pre-treated	follow EN1811 method
2917	Yes	No, new/disposable test vessel(s) were used	
3100	Yes	Yes, the previously used test vessel(s) were pre-treated	The test vessel(s) were pre-treated by being stored in a solution of 5%(m/m£©nitric acid for at least 4 hours.After acid treatment,rinse the vessel with Grade 1 water and dry.
3110	Yes	No, new/disposable test vessel(s) were used	
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	Yes, the previously used test vessel(s) were pre-treated	pre treat with nitric acid 5%
3153	Yes	No, new/disposable test vessel(s) were used	
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	Vessel was Cleaned by 10% Nitric acid For 24 hr.
3185	Yes	Yes, the previously used test vessel(s) were pre-treated	5% HNO3 cleaned the test vessel
3190	Yes	Yes, the previously used test vessel(s) were pre-treated	Soak the test vessels in nitric acid for 24H,then rinse with deionized water.
3197	Yes	Yes, the previously used test vessel(s) were pre-treated	Tests vessels are kept in %5 HNO3 for 4 hours long.
3210	Yes	No, the previously used test vessel(s) were not pre-treated	
3218	Yes	No, new/disposable test vessel(s) were used	
3225	Yes	No, new/disposable test vessel(s) were used	Nil
3228	Yes	No, new/disposable test vessel(s) were used	
3237	Yes	No, the previously used test vessel(s) were not pre-treated	

APPENDIX 4

Detailed description on how object was measured for sample #20621

lab	Please, describe as detailed as possible how you have measured and calculated the surface of the object
110	
210	
230	
310	Object is separated in 3 parts for the calculation. All parts are calculated separately. And the separate results are combined to a total sample surface area.
339	
348	The piece is divided in 3 figures, of which the surface is calculated as if they were regular solids
551	
623	Digital Caliper used to measured surface area and calculated by formulation of cuboid, rectangle, Tube surface area and half of sphere.
840	
841	
2102	The object is divided in sub-parts. The contact area of each sub-part is determined by measuring the dimensions and see it as a known geometrical figure (e.g. square).
2115	The determination was performed with calipers
2120	We used a digital caliper ruler and mathematical calculation for área for each parte and the sum of the áreas.
2129	
2132	
2135	digital caliper
2165	Suppose the cufflink was divided into 3 parts. Cylinder, cuboid and rectangle. Calculate their surface areas and add them all.
2172	breakdown into four parts for calculation, measure the surface area of the four parts separately
2184	Manually calcualtion
2201	S bottom cuboid=2*(0.794*1.606+0.0.194*1.606+0.794*0.194)=3.482cm2 S U cuboid=2*(0.774*1.617+0.301*0.134)=2.584cm2 S cylinder=2*3.14*0.2*1.43=1.796cm2 S Ball=4*3.14*0.163=0.333cm2 Stotal=8.195cm2
2216	used a 3d scanning surface area instrument
2229	three cuboids plus one globe plus one cylinder
2238	Using vernier caliper section measurement, approximate calculation
2241	calculated the area by dividing the sample into three parts: the base support part and the rotatable part.
2247	Sample was divided in small geometrical parts & sum of surface area was calculated with mathematical formula.
2250	caliper rule;1.Plate as coboid and decution of the area of solder point: 3,37cm²2.solder point as rectangle:0,104 cm² 3.Holder of pin calc. as 2 rectangles; outside: 1,148cm²; inside 1,089cm²4.Pin;2 endparts as 1 ball: 0,28 cm² and cylinder as cylinder lateral surface 1,78cm² Sum 7,771 cm²
2256	Values are in cm or sq.cm Rectangular decorate: L=1.601, W=0.786, H:0.196, (blocked part: L1=0.382, W1=0.134) U shape rod (half ellipse cross section) : L=3.705, a= 0.130, b=0.149 (w/ blocked part) Bullet backing (2 half-sphere+ cylinder): L=1.407, r= 0.205
2266	
2284	S=7.93*16.07*2+(7.93+16.07)*2.00*2+15.88*7.9*2+14.06*3.14*4.01+3.14*4*(3.31/2)2+(2.18*2-3.31)*3.14*3.31 =254.8702+96+250.904+177.0351+41.5736+10.9131 =831.3mm2 =8.31cm2
2289	Divide the sample into three parts, named the base, the supporting part and the cylinder. And calculate the surface area of the three parts respectively, then add them to get the total surface area.
2290	
2293	
2295	We measured with scanner and calliper.
2296	Using digital callipers. 3 different calculations, using variants of rectangular prism for cufflink, rectangular prism minus difference or multiple rectangular cross section for arms, and cylinder + hemisphere or capsule for end. Range 8.39-8.50.
2310	We calculate the surface area of object (rectangular& cylinder)using vernier caliper
2311	
2320	Top cuboid area- 2 rectangular top and bottom (reduced U shape joint area)+ 4 side wise rectangular U shape part-2 half cylinder + 3 rectangular + 1 circle Bottom cylinder-1 cylinder + 2 half sphere Space between Cuboid and U shape part-4 rectangular
2347	
2350	It was calculated by the sum of each part.
2352	
2357	
2363	
2365	
2366	The sample parts were divided into cylinder, sphere and cuboid, then the areas were measured and calculated by vernier caliper.
2369	
2370	This sample is divided into three parts: 1. Base cuboid: 0.793cm X 1.601cm X 2 + 0.197 X (0.793 + 1.601) X 2 = 3.482 cm2. 2.U-shaped seat: 0.295cm X (1.687cm X 2 + 1.560cm X 2) + 0.085cm X 1.560cm X 4 - 0.151cm X 0.132cm X 2 = 2.406 cm2. (The rest is shown in the remarks below)
2374	calculated by person of every surface area
2375	
2377	Use caliper to measure dimension
2378	
2379	Measured by Vernier caliper
2380	Consider this sample as 3 rectangle 2(ab+bc+ca), 3 cylinder (2ðrh) & 2 circle (ðr2). Here: a=longer length of rectangle, b=shorter length of rectangle, c=thickness of rectangle, r=radius of the cylinder & h=length of the cylinder
2381	In the cufflink we found rectangle , cylinder & circle. So we consider these law for measuring areas and finally the areas are added.
2382	a1*b1*2+（a1+b1)*c1*2+（a2*b2+a2*c2）*4+b2*c2*2+c2*a3*2+L*d1*3.14+4*3.14*r*r a:length b：width c:thickness L:cylinder height d:diameter r:radius

lab	Please, describe as detailed as possible how you have measured and calculated the surface of the object
2385	The sample was divided in several geometrical forms. For each of them, the surface area was calculated and afterwards summed up. The measurement was done with sliding caliper.
2390	Total area= base rectangle front + base rectangle big side + base rectangle small side + connector + square connector + cylinder Total area= 251.68+ 62.84+30.85+ 260.40+ 3.80+184.25= 793.82mm2 = 7.94cm2
2406	Calipers is used to calculate the surface area of different parts of cufflink, then the results are sum-up to determine the total surface area.
2410	
2425	Measured using digital slide calipers Surface area of rectangle L= 14.08mm, W= 8.62mm =(L x W) x2= 14.08x8.62x 2 = 242.74mm², Surface area of rectangle egdes L= 45.34mm W= 4.55mm = L x W = 45.34x4.55 = 206.30mm², Surface area of U clip L= 28.3mm W= 6.94mm =(L x W)x4 = 28.3x6.94x4 = 785.61mm², Surface area of cylinder L= 10.18mm D= 4.08mm = π x L x D = 3.14x10.18x4.08= 130.418mm², Surface area of half circle of cylinder D=3.09mm = (π R²/2)x2 = (3.14x 2.387/2)x2 = 7.50mm², Surface area of U clip edges L= 56.6mm W= 15.96mm = L x W = 56.6x15.96 = 903.34mm², Surface area of base U clip a= 4.10mm b= 3.35mm h=0.68mm
2426	2ab+2ah+2bh=37.60 mm², Total area=2313.51 mm² or 23.14 cm²
2429	The sample is divided into three parts, the base, the supporting part and the cylinder for measurement.
2442	Measured by calibrated digital caliper
2452	
2475	
2489	Base determined in rectangular wise including thickness & two beams determined in rectangular basis including thickness . Movable part calculated on the basis of cylindrical
2496	
2508	
2511	
2532	
2538	
2549	Rectangular Surface Area of both side = 16x8x2 = 256mm2 Rectangular 4 side area = [(16x2) +(8x2)]x2 =[32+16]x2 = 96 mm2 Side Pillar Area = 2ðrl =2x3.14x1.5x18 = 169.56 mm2 Inner Side area = 17x3x2 = 102 mm2 Rotatory Cylinder Area = 2ðrl = 2x3.14x2x14 = 175.84mm2
2560	The total surface area has been measured with the following formula: A=[2(AB+BC+CA)+2(ab+bc+ca)+πrh+2πr2
2563	
2582	Measured cufflink parts separately such as cylinder, rectangles & spheres
2590	I used the following geometrical figures: cylinder, parallelepiped and sphere
2591	We measure the surface of the piece with a vernier caliper with a precision of 0.01mm.
2624	digital caliper
2643	
2652	
2657	
2668	We consider it as two rectangular solid, and one cylinder with two hemispheres
2674	use Vernier to calculate manually
2713	
2720	Divide the sample into three parts, upper, middle and lower, measure with vernier caliper, and calculate the final area
2743	
2749	3 Determinations (different people) with slide gauge.
2804	Divided into simple geometrical form and calculated with necessary mathematical formula. S(total)=S(base)+S(supports)+S(cylinder) S(base)=2ab+2bc+2ac S(supports)=2*3.14*r1*h1+3.14*r1^2+2*3.14*r2*h2
2818	S(cylinder)=4*3.14*r3^2+2df
2864	Straighten the sample and measure the length and diameter.
2867	3 cuboids + 1 cylinder + 1 ball
2900	
2917	The cufflink was divided into 3 parts. The part of jewelry was treated like a flat quarder. The U underneath as a cylinder, which was cut lengthways. and the gag also like a cylinder ball halves at the ends
3100	The sample is divided into three parts:cylinder,cuoid and rectangle. The surface area of the sample is obtained by adding the areas of the three parts.
3110	
3116	The total surface area was composed of 3 dominant parts forming the cufflink namely the face, the post and the bullet backing. The area of each part were calculated by assuming their respective common geometrical shapes and each dimension was measured by using caliper.
3118	we use geometric approximation. The sample is divided into geometric shapes. after that we use digital caliper and calculated with geometric equation.
3153	The cufflink was measured by using a digital caliper. The toggle was calculated as a cylinder. The plate and the post were calculated as rectangular prism. The overlapped ares were subtracted.
3172	3D Scanner
3176	
3182	Using Equation L x W for 21 point. Using Equation 4 x 3.14 x r2 for 1 point. Using Equation 2 x 3.14 x r x h for 1 point.
3185	Took the sample apart,calculated each part individually, then summed them up.
3190	(Surface area of the cube) - (surface area of the hole) X 2 + (surface area of the hole wall)
3197	
3210	
3218	Bottom cuboid+ U shaped bracket+ Cylindrical- U welding point=3.482+2.726+2.095-0.124=8.18cm2
3225	The sample is divided into several parts with simpler geometry, the base, the support and the movable part. The shape, dimensions and thus area of each part are determined separately. Then, sum of area is taken as final result.
3228	base is a cuboid, outside of middle part is curve and inside is rectangle. the top part is a cylinder and two halfspheres.
3237	Calculation were made by making the parts that will hold the moving part on the base,the moving part and the edges simulate geometric shapes

APPENDIX 5

Number of participants per country

5 labs in	BANGLADESH
1 lab in	BRAZIL
4 labs in	FRANCE
8 labs in	GERMANY
1 lab in	GUATEMALA
10 labs in	HONG KONG
7 labs in	INDIA
2 labs in	INDONESIA
5 labs in	ITALY
1 lab in	MAURITIUS
1 lab in	MOROCCO
31 labs in	P.R. of CHINA
2 labs in	PAKISTAN
1 lab in	PORTUGAL
3 labs in	SOUTH KOREA
2 labs in	SPAIN
2 labs in	SRI LANKA
1 lab in	SWITZERLAND
2 labs in	TAIWAN
2 labs in	THAILAND
2 labs in	THE NETHERLANDS
2 labs in	TUNISIA
6 labs in	TURKEY
2 labs in	U.S.A.
1 lab in	UNITED KINGDOM
4 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
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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