Institute for
Interlaboratory Studies

Results of Proficiency Test
Nickel release and
Surface determination
April 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis22V03

August 2022

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 every year. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test for the determination of Nickel release and Surface.

In this interlaboratory study 113 laboratories in 27 countries registered for participation, see appendix 5 for the number of participants per country. In this report the test results of the results of the Nickel release and Surface determination 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 #22575 positive on Nickel release and one piece of a pendant labelled #22576 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

For the determination of Nickel release a batch of Nickel containing metal was selected. The batch consisted of square metal pieces with a hole in one of the corners. The pieces were solid metal, prepared from one alloy and non-coated. The dimensions of each item were approximately 2 x 2 x 0.2 cm and the hole had a diameter of approximately 0.5 cm. Three items were packed in a small plastic bag and vacuum sealed to avoid scratching of the items. 130 subsamples of three pieces each were prepared and each bag was labelled #22575. The batch for samples #22575 was used in a previous proficiency test on Nickel release and Surface determination as sample #19555 in PT iis19V03. Therefore, homogeneity of the subsamples was assumed.

For the determination of surface only a batch of pendants obtained from an (internet) shop was selected. From this batch 130 small plastic bags were filled with one piece of a pendant and this small bag was placed in an outer bag and labelled #22576. No homogeneity tests were done over the subsamples because only surface determination has been requested for this sample.

To each of the participating laboratories one sample #22575 and one sample #22576 were sent on March 23, 2022.

2.5 ANALYZES

The participants were requested to determine Nickel release on sample #22575 and to determine surface only on sample #22576.
It was 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 appendices 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 the original test results are placed under 'Remarks' in the result tables in appendices 1 and 2. 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.

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

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or

DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

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

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this 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. Therefore, the usual interpretation of z-scores is as follows:

	|z|	< 1	good
1 <	|z|	< 2	satisfactory
2 <	|z|	< 3	questionable
3 <	|z|		unsatisfactory

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. Twenty participants reported test results after the extended reporting date and one other participant did not report any test results at all.
In total 112 participants reported 331 test results numerical test results. Observed were 11 outlying test results, which is 3.3%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER SAMPLE

In this section the reported test results are discussed per sample. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. 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 in previous iis PTS. Therefore, it was decided to use a target reproducibility derived from the Horwitz equation. This target is dependent on the measured Nickel concentration, surface area and ranges from 54% at 0.3 µg Ni/cm^2/week up to 32% at 10 µg Ni/cm^2/week.

sample #22575

Nickel release: This determination may be problematic for a number of participants. Eight statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility calculated with the Horwitz equation.

Surface determination: The determination on the square piece was not problematic. Two statistical outliers were observed in the reported range of 1.5-9.7 cm^2. No official test method exists for Surface determination. Therefore, no z-scores were calculated. The variation for this sample is 0.9%.

sample #22576

Surface determination: This determination on the pendant may be problematic. Two statistical outliers were observed in the reported range of 89.9-288.7 mm^2. No official test method exists for Surface determination. Therefore, no z-scores were calculated. The variation for this sample of 13% is large but still within the range of the observed variation in previous iis PTs in which the Surface determination was evaluated (3.5-13%). It is also larger compared to the variation of the Surface determination of the much simpler shaped sample #22575 (0.9%). See also the discussion in paragraph 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 reference methods are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R(target)
Nickel release	$\mu g/cm^2/week$	101	1.10	0.46	0.49
Surface determination	cm^2	109	9.47	0.24	n.a.

Table 1: reproducibility of tests on sample #22575

Without further statistical calculations, it can be concluded that for the determination of Nickel release there is a good compliance of the group of participants with the target reproducibility.

Parameter	unit	n	average	2.8 * sd	R(target)
Surface determination	mm^2	110	218	28.2	n.a.

Table 2: reproducibility of tests on sample #22576

4.3 COMPARISON OF THE PROFICIENCY TEST OF APRIL 2022 WITH PREVIOUS PTS

	April 2022	June 2021	June 2020	June 2019	May 2018
Number of reporting laboratories	112	96	104	127	113
Number of test results	331	191	205	126	112
Number of statistical outliers	11	7	11	5	4
Percentage statistical outliers	3.3%	3.7%	5.4%	4.0%	3.6%

Table 3: comparison with previous proficiency tests

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

The performance of the determinations of the proficiency test was compared, expressed as relative standard deviation (RSD) of the PTs, see next table.

Parameter	April 2022	June 2021	June 2020	June 2019	2018-2014
Nickel release	15%	21%	29%	30%	18 - 44%
Surface determination	0.9 – 13%	0.7 – 6.9%	2.5 – 3.5%	1.1 - 7%	1.3 - 13%

Table 4: comparison of uncertainties of current PT with previous PTs

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

The uncertainty of the Surface determination of sample #22576 was large when compared to previous PTs, probably due to the complex shape of the sample.

Sample #22575 was used before in proficiency test iis19V03 as sample #19555. It is observed that the average Nickel release of sample #22575 is in line with the previous PT, see next table.

Parameter	unit	#22575			#19555		
		n	average	R(calc)	n	average	R(calc)
Nickel release	µg/cm^2/week	101	1.10	0.46	112	1.19	0.99

Table 5: comparison of sample #22575 with sample #19555

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For the Nickel release sample #22575 the following can be summarized from the various analytical details provided by the participants (these are given in appendix 2 and 3):

- About 95% of the reporting laboratories mentioned to be accredited for the determination of Nickel release.
- About 65% of the reporting participants have used new or disposable test vessels, while about 30% of the reporting participants done a pre-treatment of the test vessels. Remarkably, about 5% of the reporting participants have not done any pre-treatment of the previously used test vessels.

- About 70% of the reporting participants used a ratio of approximately 1 mL test solution per cm^2 sample surface area.

For the Surface determination sample #22576 was requested for a detailed description on how the surface area was measured and calculated. A variety of methods was given by about 70% of the reporting participants, see appendix 4 for the answers given.

5 DISCUSSION

sample #22575
The variation on the Nickel release results for the individual laboratories was large; with an RSD from 0.3% up to 64.3%. Only 44 participants had an RSDr in agreement with the target repeatability standard deviation of 4%, estimated from EN1811:11+A1:15 (33.3% / 2.8 / 3). Remarkedly, when evaluating the test results of only those 44 participants the average and variation are not significant different from the total group. See appendix 1 for the summary of evaluation.

Test method EN1811:11+A1:15 prescribes the amount of initial test solution to be used to be 1 mL per cm^2 surface area. 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, the average and variation are not significant different from the total group. See appendix 1 for the summary of evaluation.

sample #22576
The variation on the determination of the Surface area of the pendant was larger compared to previous iis PTs. This is most likely due to the complex shape of the sample. The pendant consists of a ring and body. The body was of a lightning shape which contains a red colored part on the front, see figure 1 below.

Figure 1: picture of sample #22576 (front and back)

When all reported test results of the determination of the pendant are taken into account the RSD is 13%. However, from the description given by the participants on how the Surface area was measured and calculated it was clear that several methods were used. It was also clear that the participants made different decisions of which parts of the pendant need to be included in the calculation of the Surface area of the pendant. Some participants included the red colored part in the calculation of the Surface area, while others did not take the red part into the calculation. The same is observed about the ring or about the edge of the pendant. From all the descriptions given we have tried to extract the results which were calculated with the red colored part and also those without the red colored part, see appendix 1.
Based on the extraction done by iis the results which include the red colored part shows a decrease in RSD compared to the RSD of all results given by the respondents (5% vs 13%). The results without the red colored part shows an RSD more in line with the RSD of all results (11% vs 13%).

6 CONCLUSION

It can be concluded that the majority of the participants had no problems with the determination of Nickel release.

For the determination of the Surface area of a complex shape it is important that it is clear which parts of the sample should be included in the calculation of the area.

Each participating laboratory will have 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.

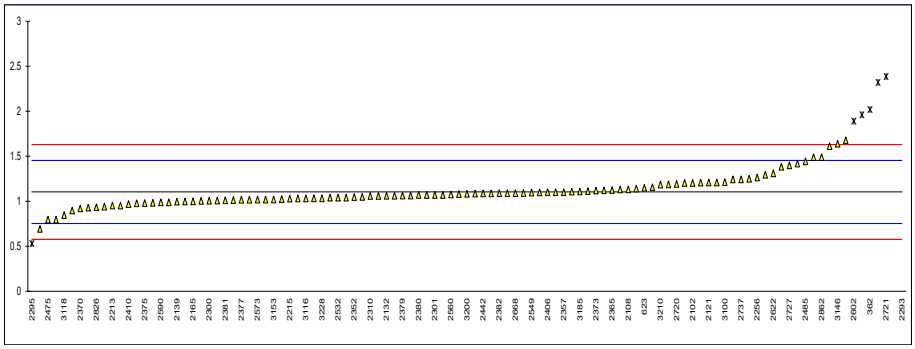
APPENDIX 1

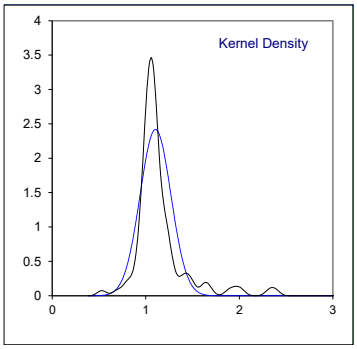
Determination of Nickel release on sample #22575; average result of three replicates in µg/cm²/week

lab	method	value	mark	z(targ)	remarks
210	EN1811 + A1	1.07	C	-0.19	first reported 0.44
339		-----		-----	
362		2.02	R(0.01)	5.22	
551	EN1811 + A1	1.0869		-0.10	
623	EN1811	1.15		0.26	
2102		1.2042		0.57	
2108	EN1811 + A1	1.132		0.16	
2115	EN1811	1.49		2.20	
2121	EN1811	1.21	C	0.61	first reported 3.63
2132	EN1811 + A1	1.062		-0.24	
2138	EN1811 + A1	1.10		-0.02	
2139	EN1811	0.996		-0.61	
2159	EN1811	0.998		-0.60	
2165	EN1811	0.999		-0.60	
2184	EN1811 + A1	0.984		-0.68	
2201	EN1811	1.189		0.49	
2213	EN1811	0.953		-0.86	
2215	EN1811	1.030		-0.42	
2216		-----		-----	iis calc. average 1.77: z(targ) = 3.79
2238	EN1811	1.42		1.80	
2256	EN1811	1.264		0.91	
2289	EN1811	1.204		0.57	
2290	EN1811 + A1	0.979		-0.71	
2293	EN1811 + A1	4.893366667	C,R(0.01)	21.60	first reported 4.2266
2295	EN1811	0.53	R(0.01)	-3.27	
2300	EN1811 + A1	1.007		-0.55	
2301	EN1811	1.07	C	-0.19	first reported 1.70
2310	EN1811	1.06		-0.25	
2311	EN1811 + A1	1.04		-0.36	
2330		-----		-----	iis calc. average 2.443: z(targ) = 7.63
2347	EN1811 + A1	1.05		-0.31	
2350	EN1811 + A1	1.295		1.09	
2352	EN1811 + A1	1.048		-0.32	
2357	EN1811 + A1	1.10		-0.02	
2363	EN1811 + A1	1.121		0.10	
2365	EN1811 + A1	1.125		0.12	
2366	EN1811 + A1	1.033		-0.40	
2369	EN1811 + A1	1.064		-0.23	
2370	EN1811	0.921		-1.04	
2373	EN1811 + A1	1.119		0.09	
2375	EN1811	0.98		-0.71	
2377	EN1811	1.018		-0.49	
2378	GB/T30158	1.08		-0.14	
2379	EN1811 + A1	1.063	C	-0.23	first reported 1.910
2380	EN1811 + A1	1.070		-0.19	
2381	EN1811	1.0114		-0.53	
2382	EN1811 + A1	1.09		-0.08	
2385	EN1811	1.679		3.28	
2390	EN1811 + A1	1.005		-0.56	
2406	EN1811 + A1	1.100		-0.02	
2410	EN1811	0.97		-0.76	
2425	EN1811 + AC	1.21		0.61	
2426	EN1811 + A1	0.930		-0.99	
2429		1.208		0.59	
2442	EN1811 + A1	1.088		-0.09	
2462	EN1811	1.014		-0.51	
2475	EN1811	0.797		-1.75	
2485	EN1811 + A1	1.445		1.94	
2489	EN1811	1.06		-0.25	
2495	EN1811 + A1	1.243		0.79	
2511	EN1811	1.14		0.21	
2515	EN1811	1.062		-0.24	
2532	EN1811 + A1	1.04	C	-0.36	first reported 2.0
2538	EN1811 + A1	1.9604	R(0.01)	4.88	
2549	EN1811 + A1	1.097		-0.04	
2560		1.076		-0.16	
2573	EN1811	1.020		-0.48	
2582	EN1811 + A1	2.3212	C,R(0.01)	6.94	first reported 1.9222
2590	EN1811	0.9899		-0.65	
2591	EN1811	0.69352		-2.34	
2602	EN1811	1.89	C,R(0.01)	4.48	first reported 2.25
2622	EN1811	1.31		1.18	
2624		-----		-----	
2637	EN1811	4.67	R(0.01)	20.33	

lab	method	value	mark	z(targ)	remarks
2649	EN1811	1.04		-0.36	
2652	EN1811	1.090		-0.08	
2668	EN1811	1.09		-0.08	
2674	EN1811 + A1	1.018		-0.49	
2678	EN1811 + A1	1.156		0.30	
2720	EN1811 + A1	1.193		0.51	
2721	In house	2.386	R(0.01)	7.31	
2727	EN1811 + A1	1.400		1.69	
2737	EN1811	1.24373		0.80	
2741	EN1811	0.939		-0.94	
2743	EN1811 + A1	0.797		-1.75	
2758	EN1811	1.031		-0.42	
2818	EN1811	1.008		-0.55	
2826	EN1811 + A1	0.934		-0.97	
2827	EN1811	1.099		-0.03	
2829	EN1811 + A1	1.025	C	-0.45	first reported 1.714
2858	EN1811	1.091		-0.07	
2862	EN1811	1.49		2.20	
2864	EN1811 + A1	1.07		-0.19	
2959	EN1811 + A1	1.113		0.05	
3100		1.2133		0.62	
3110		0.953		-0.86	
3116	EN1811 + A1	1.0318		-0.41	
3118	EN1811 + A1	0.8466	C	-1.47	first reported 2.1164
3146	EN1811 + A1	1.640		3.06	
3153	EN1811 + A1	1.021		-0.47	
3160	EN1811 + A1	1.13		0.15	
3172	EN1811 + AC	1.088		-0.09	
3182	EN1811	1.612	C	2.90	first reported 1.726
3185		1.109		0.03	
3197	EN1811 + A1	0.99	C	-0.65	first reported 1.98
3200	EN1811	1.083		-0.12	
3209	EN1811	1.02		-0.48	
3210	EN1811	1.1871		0.47	
3214	EN1811 + A1	1.382		1.59	
3218	EN1811	1.108		0.02	
3228		1.033		-0.40	
3230	In house	0.8966		-1.18	
3246		1.250		0.83	

			Only RSD <4% between replicates		Only with ratio 1:1	
normality	not OK		not OK		not OK	
n	101		42		63	
outliers	8		2		4	
mean (n)	1.104		1.088		1.105	
st.dev. (n)	0.1650	RSD=15%	0.1249	RSD=11%	0.1660	RSD=15%
R(calc.)	0.462		0.350		0.465	
st.dev.(Horwitz)	0.1754		0.1733		0.1756	
R(Horwitz)	0.491		0.485		0.492	
compare R(EN1811:11+A1:15)	0.368					



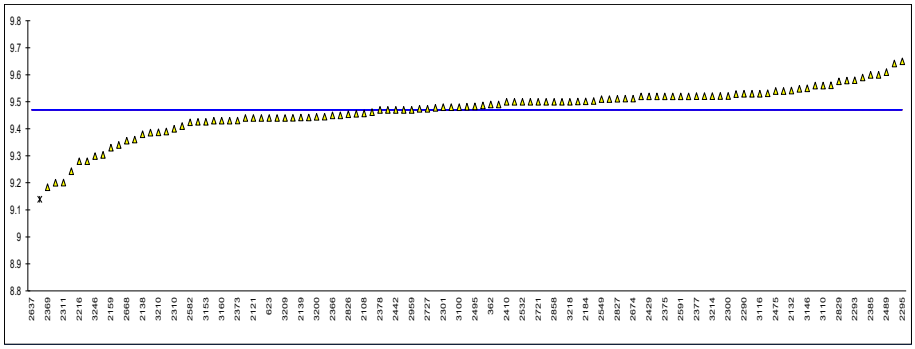


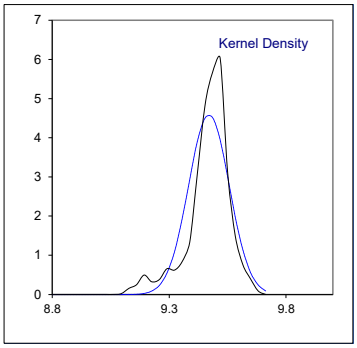
Determination of Surface on sample #22575; results in cm^2

lab	method	value	mark	z(targ)	remarks
210		9.44		-----	
339		-----		-----	
362		9.49		-----	
551		9.36036		-----	
623		9.44		-----	
2102		9.34		-----	
2108		9.457		-----	
2115		9.49		-----	
2121		9.44		-----	
2132		9.542		-----	
2138		9.38		-----	
2139		9.4420		-----	
2159		9.33		-----	
2165		9.480		-----	
2184		9.502		-----	
2201		9.50		-----	
2213		9.5289		-----	
2215		9.590		-----	
2216		9.28		-----	
2238		9.41		-----	
2256		9.482		-----	
2289		9.52		-----	
2290		9.53		-----	
2293		9.58		-----	
2295		9.65		-----	
2300		9.522		-----	
2301		9.48		-----	
2310		9.4		-----	
2311		9.201		-----	
2330		9.5787		-----	
2347		9.44		-----	
2350		9.52		-----	
2352		9.386		-----	
2357		9.45		-----	
2363		9.39		-----	
2365		9.426		-----	
2366		9.45		-----	
2369		9.184		-----	
2370		9.47		-----	
2373		9.431		-----	
2375		9.52		-----	
2377		9.521		-----	
2378		9.47		-----	
2379		9.522		-----	
2380		9.512		-----	
2381		9.561		-----	
2382		9.56		-----	
2385		9.6		-----	
2390		9.441		-----	
2406		9.548		-----	
2410		9.5		-----	
2425		9.2		-----	
2426		9.28		-----	
2429		9.52		-----	
2442		9.470		-----	
2462		9.462		-----	
2475		9.54		-----	
2485		9.52		-----	
2489		9.61		-----	
2495		9.483		-----	
2511		9.47		-----	
2515		9.445		-----	
2532		9.5		-----	
2538		9.4866666		-----	*)
2549		9.51		-----	
2560		9.503		-----	
2573		9.642		-----	
2582		9.4243		-----	
2590		9.4559		-----	
2591		9.52		-----	
2602		9.52		-----	
2622		9.50		-----	
2624		-----		-----	
2637		1.49	R(0.01)	-----	
2649		9.54		-----	

lab	method	value	mark	z(targ)	remarks
2652		9.478		-----	
2668		9.356		-----	
2674		9.512		-----	
2678		9.501		-----	
2720		9.474		-----	
2721		9.50		-----	
2727		9.474		-----	
2737		9.50		-----	
2741		9.6		-----	
2743		9.532		-----	
2758		9.43		-----	
2818		9.521		-----	
2826		9.4544		-----	
2827		9.511		-----	
2829		9.576		-----	
2858		9.500		-----	
2862		9.5		-----	
2864		9.53		-----	
2959		9.47		-----	
3100		9.4800		-----	
3110		9.56		-----	
3116		9.53		-----	
3118		9.2431		-----	
3146		9.55		-----	
3153		9.426		-----	
3160		9.43		-----	
3172		9.139	C,R(0.05)	-----	first reported 238.6
3182		9.44		-----	
3185		9.442		-----	
3197		9.43		-----	
3200		9.444		-----	
3209		9.44		-----	
3210		9.387		-----	
3214		9.521		-----	
3218		9.500		-----	
3228		9.51		-----	
3230		9.3036		-----	
3246		9.299		-----	
	normality	suspect			
	n	109			
	outliers	2			
	mean (n)	9.470			
	st.dev. (n)	0.0869	RSD=0.9%		
	R(calc.)	0.243			

*) Lab 2538 noted: the items had to be migrated immediately, therefore the surface of one item was measured (and calculated) in wrapped condition for calculation the sweat-solution

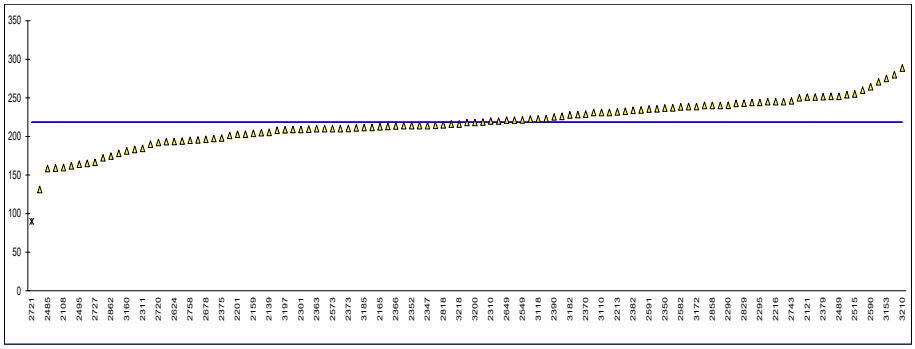


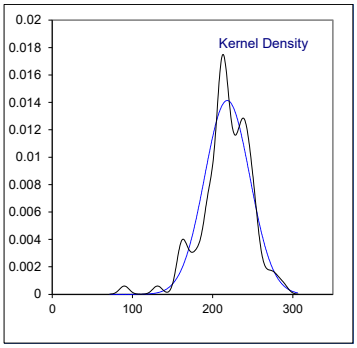


Determination of Surface on sample #22576; results in mm^2

lab	method	value	mark	z(targ)	iis extracted from Q&A + comments: value with red part	value without red part	remarks
210		183	C	-----	-----	-----	reported 1.83 mm^2
339		-----		-----	-----	-----	
362		161.9		-----	-----	-----	
551		131.27		-----	189.8985 G5	131.27	
623		193.18		-----	-----	-----	
2102		242.71		-----	-----	-----	
2108		159.7		-----	-----	-----	
2115		208		-----	-----	-----	
2121		251		-----	-----	-----	
2132		270.74		-----	-----	-----	
2138		201.31		-----	-----	-----	
2139		205.236		-----	-----	-----	
2159		204		-----	-----	-----	
2165		212.5		-----	-----	212.5	
2184		221.2		-----	-----	-----	
2201		202.8		-----	-----	202.8	
2213		231.85		-----	231.85	171	
2215		213		-----	-----	-----	
2216		245		-----	-----	-----	
2238		203		-----	-----	-----	
2256		213.9		-----	-----	213.9	
2289		189.82		-----	-----	189.82	
2290		240.3		-----	-----	-----	
2293		209		-----	-----	209	
2295		244		-----	244	184	
2300		178.11		-----	-----	-----	
2301		209		-----	-----	-----	
2310		220		-----	-----	-----	
2311		184.62		-----	-----	-----	
2330		279.950		-----	-----	-----	
2347		214		-----	-----	-----	
2350		236.88		-----	-----	-----	
2352		214.0		-----	-----	-----	
2357		210		-----	-----	-----	
2363		210		-----	-----	-----	
2365		214.0		-----	-----	-----	
2366		213.6		-----	-----	-----	
2369		205		-----	-----	-----	
2370		229		-----	-----	-----	
2373		210.3		-----	-----	210.3	
2375		198		-----	-----	-----	
2377		234.3	C	-----	-----	-----	reported 2.343 mm^2
2378		218		-----	-----	-----	
2379		251.478		-----	-----	-----	
2380		231.1		-----	-----	-----	
2381		232.594		-----	-----	-----	
2382		234		-----	-----	-----	
2385		165		-----	-----	165	
2390		225.33		-----	-----	-----	
2406		222.92		-----	222.92	-----	
2410		250		-----	-----	-----	
2425		245.0		-----	-----	-----	
2426		251.94		-----	-----	-----	
2429		197.3	C	-----	-----	197.3	reported 1.973 mm^2
2442		237.060		-----	237.060	178.06	
2462		218.7		-----	-----	-----	
2475		211		-----	-----	-----	
2485		158.504		-----	-----	-----	
2489		252	C	-----	-----	-----	first reported 25.2
2495		164		-----	-----	164	
2511		238.6		-----	-----	-----	
2515		255.097		-----	-----	-----	
2532		220		-----	-----	-----	
2538		228.27		-----	-----	-----	
2549		221.320		-----	-----	-----	
2560		240.020		-----	240.020	179.48	
2573		210		-----	-----	-----	
2582		238.33		-----	238.33	-----	
2590		264.4		-----	-----	-----	
2591		235.51		-----	-----	-----	
2602		243.9		-----	243.9	-----	
2622		251		-----	251	200	

lab	method	value	mark	z(targ)	iis extracted from Q&A + comments: value with red part	value without red part	remarks
2624		193.4		-----	-----	-----	
2637		-----		-----	-----	-----	
2649		221.09		-----	221.09	-----	
2652		216		-----	-----	-----	
2668		235.86		-----	-----	-----	
2674		214.31		-----	-----	-----	
2678		196.30		-----	-----	-----	
2720		192.18		-----	-----	192.18	
2721		89.9	R(0.01)	-----	-----	89.9 G1	
2727		166.421		-----	-----	-----	
2737		209.42		-----	-----	209.42	
2741		194.1	C	-----	-----	-----	first reported 1941
2743		245.959		-----	-----	-----	
2758		195	C	-----	-----	195	first reported 19.51
2818		215.0		-----	-----	-----	
2826		259.96		-----	259.96	-----	
2827		240.165		-----	-----	-----	
2829		243.1		-----	-----	-----	
2858		240.1		-----	240.1	182.5	
2862		174.5		-----	-----	174.5	
2864		231		-----	-----	-----	
2959		245		-----	-----	-----	
3100		195.540303		-----	-----	195.540303	
3110		231		-----	-----	-----	
3116		254.12		-----	-----	-----	
3118		222.7501		-----	-----	-----	
3146		226.2		-----	-----	-----	
3153		275		-----	-----	-----	
3160		181.26		-----	-----	181.26	
3172		238.6		-----	238.6	185.58	
3182		227.85		-----	267.79	227.85	
3185		211.4		-----	-----	-----	
3197		208.6		-----	-----	208.6	
3200		218		-----	-----	-----	
3209		222.62		-----	-----	-----	
3210		288.67		-----	-----	-----	
3214		158.971		-----	-----	158.971	
3218		216		-----	-----	216	
3228		210		-----	-----	-----	
3230		172.257		-----	-----	172.257	
3246		211.6	C	-----	-----	-----	fr. 2.116 mm^2
	normality	OK			OK	not OK	
	n	110			13	28	
	outliers	1			1	1	
	mean (n)	218.3	RSD=13%		241.3 RSD=5%	186.1 RSD=11%	
	st.dev. (n)	28.22			13.01	21.19	
	R(calc.)	79.0			36.4	59.3	





APPENDIX 2

Determination of Nickel release and some Analytical Details on subsamples #22575

lab	value plate 1 (µg/cm2/week)		value plate 2 (µg/cm2/week)		value plate 3 (µg/cm2/week)		RSDr (%) calc. by iis		volume test solution (mL)		ratio volume vs. area (mL/cm²)
210	1.03	C	1.11	C	1.08	C	3.8		-----		
339	-----		-----		-----		-----		-----		
362	2.12		2.04		1.91		5.2		10		
551	0.7461		1.2473		1.2673		27.2		12		1.28 mL/cm²
623	0.6841		0.7729		2.0074		64.3		20		1:1
2102	1.2138		1.1640		1.2349		3.0		10		1:1
2108	1.053		2.345		1.211		62.3		10		approx. 2,5 ml/cm2
2115	2.10		1.57		0.79		44.2		12.5		1:1
2121	0.90		1.26		1.47		23.8		20		Around 2.1 mL/cm²
2132	0.8614		1.150		1.174		16.4		9.90		1:0.964
2138	1.09		1.10		1.11		0.9		10		1:1
2139	1.089		0.987		0.911		9.0		10		1.059
2159	1.138		0.952		0.905		12.3		22.5		25/10 ml/cm²
2165	0.945		1.045		1.008		5.1		10		Approximately 1:1
2184	0.982		1.016		0.953		3.2		10		1:1
2201	1.188		1.197		1.184		0.6		9.5		1:1
2213	0.98		0.92		0.96		3.2		12		1
2215	1.024		1.026		1.041		0.9		16		16:9.59
2216	1.92		1.81		1.59		9.5		10		~1:1
2238	1.35		1.70		1.22		17.5		10		1.06
2256	1.218		1.311		1.262		3.7		15		1.58
2289	1.219		1.241		1.152		3.9		9.5		1:1
2290	0.918		1.007		1.013		5.4		-----		
2293	5.0574		4.7166	C	4.9061		3.5		9.58		1:1
2295	0.51		0.57		0.52		6.1		15		1.55
2300	0.932		1.649		0.440		60.4	*)	28	*)	Almost 1:4
2301	1.55		1.68		1.88		15.5		10		2:1
2310	1.06		1.1		1.02		3.8		10		1:1
2311	1.07		1.01		1.04		2.9		9.5		1:1
2330	1.885		3.492		1.952		37.2		10		1:1
2347	1.03		1.04		1.07		2.0		9.44		1:1
2350	0.823		1.248		1.814		38.4		10		20/9.52 =2.101 mL/cm²
2352	0.978		1.059		1.107		6.2		9.39		1:1
2357	1.228		1.124		0.950		12.8		-----		
2363	1.145		1.089		1.129		2.6		9.39		1:1
2365	1.154		1.215		1.005		9.6		9.43		1:1
2366	0.979		1.016		1.104		6.2		-----		
2369	1.078		1.066		1.048		1.4		-----		
2370	0.988		0.865		0.909		6.8		20		2mL/1cm2
2373	1.112		1.121		1.124		0.6		9.43		1:1
2375	0.96		0.94		1.05		6.0		9.52		1:1
2377	1.008		0.992		1.054		3.2		10		1.05 mL/cm2
2378	1.008		1.052		1.179		8.2		9.5		1:1
2379	0.789	C	1.625	C	0.774	C	45.8		10		1:1
2380	1.022		1.085		1.104		4.0		12		1:1 (ml)
2381	1.0030		0.9848		1.0465		3.1		10		1:1
2382	1.07		1.09		1.11		1.8		9.56		1:1
2385	1.576		1.962		1.500		14.8		12		1.25
2390	1.028		0.985		1.001		2.2		9.4		1:1
2406	0.816		1.126		1.359		24.8		10		1:1
2410	1.25		0.85		0.81		25.1		15		1 : 1.6
2425	1.19		1.21		1.23		1.7		9		1:1
2426	0.930		0.950		0.910		2.2		9.3		1ml test solution per cm2
2429	1.179		1.257		1.188		3.5		10		1.05:1
2442	0.930		1.098		1.235		14.0		9.5		1:1
2462	1.014		0.976		1.051		3.7		10		10.0 ml: 9.46 cm2
2475	1.250		0.500		0.641		50.0		9.52		1
2485	1.100		2.478		0.758		63.0		10		around 1:1
2489	1.09		1.09		1.02		3.8		10		5:10
2495	1.644		1.087		0.999		28.1		20		2
2511	1.23		1.05		1.15		7.9		-----		
2515	1.061		1.102		1.024		3.7		12		20/9.445
2532	1.07	C	1.04	C	1.02	C	2.4		20		25ml test solution /9.5 cm2
2538	1.5894		2.0975		2.1944		16.6		10		10 ml / 9,94 cm²
2549	0.998		1.232		1.061		11.0		10		10 ml/9.51 cm2
2560	1.076		1.287		0.866		19.6		12		1.2 : 1
2573	1.142		1.017		0.901		11.8		10		1:1
2582	1.9211		1.9317		1.9139		0.4		9		10 mL per (5-10)cm²
2590	0.9666		1.0639		0.9391		6.6		20		2 mL/cm^2
2591	0.79285		0.93706		0.35066		44.1		9.5		1/1
2602	-----	W	1.74		2.03		10.8		10		~ 1mL / 1cm2
2622	0.963		1.26		1.72		29.1		10		1:1
2624	-----		-----		-----		-----		-----		
2637	5.47		3.84		4.71		17.5		-----		

lab	value plate 1 (µg/cm2/week)	value plate 2 (µg/cm2/week)	value plate 3 (µg/cm2/week)	RSDr (%) calc. by iis	volume test solution (mL)	ratio volume vs. area (mL/cm²)
2649	1.00	1.04	1.08	3.8	12	1.25:1
2652	1.091	0.971	1.209	10.9	-----	
2668	1.09	1.08	1.11	1.4	10.5	1:1
2674	1.061	1.053	0.941	6.6	13	1.37:1
2678	1.120	1.140	1.210	4.1	9.50	1.00
2720	1.292	1.172	1.114	7.6	9.47	1:1
2721	2.048	2.548	2.561	12.3	23	2.4
2727	1.541	1.285	1.373	9.3	22	2.4
2737	1.174	1.367	1.190	8.6	9.5	1 : 1
2741	0.951	0.923	0.944	1.6	12	1.25
2743	0.963	0.724	0.703	18.1	20	2 ml/cm2
2758	1.028	0.926	1.138	10.3	10	1
2818	1.189	1.001	0.834	17.6	9.52	9.52mL:9.52cm2
2826	0.990	1.00	0.813	11.3	10	1.06mL/cm2
2827	1.067	1.112	1.118	2.5	9.5	1:1
2829	1.770	1.710	1.663	5.2	11.00	1.1
2858	0.821	1.321	1.132	23.1	10	1:1
2862	1.38	1.595	2.39	10.2	14	14ml for 9,5 cm2
2864	1.13	1.07	1.01	5.6	10	1
2959	1.090	1.124	1.126	1.8	-----	
3100	1.2397	1.1563	1.2439	4.1	9.48 *)	1:1
3110	0.9	0.81	1.15	18.5	-----	
3116	1.0850	0.9955	1.0150	4.6	10	1
3118	0.8233 C	0.8401 C	0.8764 C	3.2	10	1:1
3146	2.140	1.200	1.580	28.8	9.6	ratio 1:1
3153	1.024	1.079	0.961	5.8	9.4	1:1
3160	1.52	1.18	0.69	36.9	10	1.06
3172	1.2250	1.2540	0.7854	24.1	-----	
3182	1.631 C	1.610 C	1.608 C	0.8	9.44	1:1
3185	1.119	1.110	1.097	1.0	9.5	1:1
3197	1.53	2.17	2.20	38.2	9.43	1:1
3200	1.149	1.041	1.059	5.3	9.44	1:1
3209	1.05	0.97	1.03	4.1	10.0	1:1
3210	1.2224	1.4560	0.8830	24.3	30	3.2
3214	1.253	1.539	1.353	10.5	9.52	1:1
3218	1.146	1.085	1.094	3.0	15	15mL/9.5cm2
3228	1.04	1.01	1.05	2.0	9.51	1:1
3230	0.90015	0.8948	0.8949	0.3	15	1.61
3246	1.208	1.286	1.258	3.2	15	1.66

Lab 210: first reported 0.42, 0.45 and 0.44 respectively
Lab 2293: first reported 2.7166
Lab 2379: first reported 2.062, 1.652 and 2.015 respectively
Lab 2532: first reported 2.2, 2.4 and 1.4 respectively
Lab 2602: test result withdrawn, reported 2.98
Lab 3118: first reported 2.0582, 2.1001 and 2.1909 respectively
Lab 3182: first reported 1.744, 1.752 and 1.683

*)
Lab 2300 noted: 3 samples are showing variation in Nickel release value, though the analysis is conducted at the same conditions. For 1st sample the volume is taken 30ml, For 2nd sample the volume is taken 28ml and for 3rd sample the volume is taken 26ml. The Volume is adjusted just to dip the sample entirely. Because the sample is kept in suspended position without touching the bottom and the wall of the vessel.
Lab 3100 noted: #22575-A:9.46mL; #22575-B:9.56mL; #22575-C:9.43mL

APPENDIX 3

Other reported Analytical Details for sample #22575

lab	ISO17025 accred.	pre-treat test vessel	pre-treatment procedure
210	Yes	---	
339	---	---	
362	Yes	No, new/disposable test vessel(s) were used	
551	Yes	Yes, the previously used test vessel(s) were pre-treated	Glassware washed with 10% neutral Extran detergent, left in a 10% HNO3 bath for a period of 24 hours, after time, washed with deionized water.
623	Yes	Yes, the previously used test vessel(s) were pre-treated	soak in acid bath 5%
2102	Yes	Yes, the previously used test vessel(s) were pre-treated	Vessels are filled with nitric acid (5%), after 4 hours the vessel is rinsed with deionized water and dried.
2108	Yes	No, new/disposable test vessel(s) were used	
2115	Yes	No, new/disposable test vessel(s) were used	
2121	Yes	No, new/disposable test vessel(s) were used	
2132	Yes	No, new/disposable test vessel(s) were used	
2138	Yes	No, new/disposable test vessel(s) were used	
2139	Yes	No, new/disposable test vessel(s) were used	
2159	Yes	No, new/disposable test vessel(s) were used	
2165	Yes	No, new/disposable test vessel(s) were used	
2184	Yes	No, the previously used test vessel(s) were not pre-treated	
2201	Yes	Yes, the previously used test vessel(s) were pre-treated	The vessel and holder were pre-treated by stored 10% nitric acid solution over night, after acid cleaning, rinse the vessel and holder with deionised water thoroughly and dry
2213	Yes	No, new/disposable test vessel(s) were used	
2215	Yes	Yes, the previously used test vessel(s) were pre-treated	Stored in 5% nitric acid for 4 H, rinse and holder with deionised water and dry.
2216	Yes	Other	Soaked new test vessels in 5% HNO3 for 4 hours
2238	Yes	No, new/disposable test vessel(s) were used	
2256	Yes	No, new/disposable test vessel(s) were used	
2289	---	---	
2290	Yes	---	
2293	Yes	No, new/disposable test vessel(s) were used	
2295	Yes	No, new/disposable test vessel(s) were used	
2300	Yes	Yes, the previously used test vessel(s) were pre-treated	The vessel is treated with 5% Nitric acid for 4 hours. After acid cleaning the vessel is rinsed with water and dried.
2301	Yes	Yes, the previously used test vessel(s) were pre-treated	pre-treated in a solution of nitric acid 5% for 4 hours. After acid treatment, the vessel was rinsed with deionized water and dry
2310	Yes	No, new/disposable test vessel(s) were used	Dil.nitric acid and then rinsed with deionized water and dry the vessel
2311	Yes	Yes, the previously used test vessel(s) were pre-treated	Pretreat with 5% Nitric acid, rinsed with deionized water and dried.
2330	No	Yes, the previously used test vessel(s) were pre-treated	Immerse in 10% Nitric acid over night
2347	Yes	No, new/disposable test vessel(s) were used	
2350	Yes	No, new/disposable test vessel(s) were used	
2352	Yes	No, new/disposable test vessel(s) were used	
2357	---	---	
2363	Yes	Yes, the previously used test vessel(s) were pre-treated	use 5% HNO3 stay for 4 hour
2365	Yes	No, new/disposable test vessel(s) were used	
2366	Yes	---	
2369	Yes	---	
2370	Yes	No, new/disposable test vessel(s) were used	
2373	Yes	No, new/disposable test vessel(s) were used	
2375	---	---	
2377	Yes	No, new/disposable test vessel(s) were used	
2378	Yes	No, new/disposable test vessel(s) were used	
2379	Yes	Yes, the previously used test vessel(s) were pre-treated	5 % HNO3 4 hours
2380	Yes	Yes, the previously used test vessel(s) were pre-treated	Rinse with tape water & wash with detergent, then rinse with tape water 5 times & with deionized water 2 times. Sock in acid bath (5% Nitric acid) for over night. Rinse with deionized water thoroughly 10 times. Dry the glassware.
2381	Yes	Yes, the previously used test vessel(s) were pre-treated	All test vessels are pretreated by 5% Nitric acid for 4 hours and then rinsed with deionized water and dry.
2382	Yes	No, the previously used test vessel(s) were not pre-treated	
2385	Yes	No, new/disposable test vessel(s) were used	
2390	Yes	No, the previously used test vessel(s) were not pre-treated	
2406	Yes	No, new/disposable test vessel(s) were used	
2410	Yes	Yes, the previously used test vessel(s) were pre-treated	Pre-treatment by 5 % nitric acid for at least 4h.
2425	Yes	No, new/disposable test vessel(s) were used	

lab	ISO17025 accred.	pre-treat test vessel	pre-treatment procedure
2426	Yes	No, new/disposable test vessel(s) were used	
2429	Yes	Yes, the previously used test vessel(s) were pre-treated	Pre-treaded 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	
2462	Yes	No, new/disposable test vessel(s) were used	
2475	Yes	Yes, the previously used test vessel(s) were pre-treated	HNO3 5% during 4H; After acid cleaning, rinse the vessel with deionised water and dry
2485	Yes	Yes, the previously used test vessel(s) were pre-treated	as described in the norm, the vessels have been rinsed with diluted HNO3.
2489	Yes	No, new/disposable test vessel(s) were used	
2495	Yes	No, new/disposable test vessel(s) were used	
2511	---	---	
2515	Yes	No, new/disposable test vessel(s) were used	
2532	Yes	No, new/disposable test vessel(s) were used	
2538	Yes	No, new/disposable test vessel(s) were used	---
2549	Yes	No, new/disposable test vessel(s) were used	
2560	Yes	Yes, the previously used test vessel(s) were pre-treated	finally rinsed with grade-I water.
2573	---	---	
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	
2602	Yes	No, new/disposable test vessel(s) were used	---
2622	Yes	No, the previously used test vessel(s) were not pre-treated	
2624	---	---	
2637	Yes	No, new/disposable test vessel(s) were used	
2649	Yes	No, new/disposable test vessel(s) were used	
2652	Yes	Yes, the previously used test vessel(s) were pre-treated	
2668	Yes	No, new/disposable test vessel(s) were used	
2674	Yes	Yes, the previously used test vessel(s) were pre-treated	5%HNO3 steep 4 hours
2678	No	No, new/disposable test vessel(s) were used	
2720	Yes	Yes, the previously used test vessel(s) were pre-treated	Soak in 5% nitric acid for 4 hours
2721	Yes	Yes, the previously used test vessel(s) were pre-treated	pickled overnight in 5 % HNO3
2727	Yes	No, new/disposable test vessel(s) were used	
2737	Yes	Yes, the previously used test vessel(s) were pre-treated	Lids and vessels are stored in a solution of nitric acid at least 4 hours
2741	Yes	No, new/disposable test vessel(s) were used	
2743	Yes	No, new/disposable test vessel(s) were used	
2758	No	No, new/disposable test vessel(s) were used	
2818	Yes	No, new/disposable test vessel(s) were used	
2826	Yes	No, new/disposable test vessel(s) were used	
2827	Yes	No, new/disposable test vessel(s) were used	
2829	Yes	No, new/disposable test vessel(s) were used	
2858	Yes	No, new/disposable test vessel(s) were used	
2862	Yes	No, new/disposable test vessel(s) were used	
2864	Yes	No, new/disposable test vessel(s) were used	
2959	---	---	
3100	Yes	Yes, the previously used test vessel(s) were pre-treated	the vessel and holder shall be pre-treated by being stored in a solution of diluted nitric acid (5.11) for at least 4 h. After acid cleaning, rinse the vessel and holder with deionised water and dry
3110	---	---	
3116	Yes	Yes, the previously used test vessel(s) were pre-treated	Soaked the vessels in 5% nitric acid solution for 4 hours and then rinsed with deionized water
3118	Yes	No, new/disposable test vessel(s) were used	-
3146	Yes	No, new/disposable test vessel(s) were used	
3153	Yes	No, new/disposable test vessel(s) were used	
3160	No	No, new/disposable test vessel(s) were used	
3172	---	---	
3182	No	Yes, the previously used test vessel(s) were pre-treated	10% HNO3
3185	Yes	Yes, the previously used test vessel(s) were pre-treated	Test vessels were pre-treated by being stored in a solution of nitric acid with mass fraction of 5% for at least 4 hours. After acid treatment, rinse the vessel with ultrapure water and dry.
3197	Yes	Yes, the previously used test vessel(s) were pre-treated	The tests vessels are pre-treated in %5 HNO3 for 4 hours long.
3200	Yes	No, new/disposable test vessel(s) were used	
3209	Yes	No, new/disposable test vessel(s) were used	
3210	Yes	No, the previously used test vessel(s) were not pre-treated	

lab	ISO17025 accred.	pre-treat test vessel	pre-treatment procedure
3214	Yes	Yes, the previously used test vessel(s) were pre-treated	Soak all vessels in 50% nitric acid for 24h at room temperature, and then rinse thoroughly with D.I. Water
3218	Yes	No, new/disposable test vessel(s) were used	
3228	Yes	No, new/disposable test vessel(s) were used	
3230	Yes	No, new/disposable test vessel(s) were used	
3246	Yes	No, new/disposable test vessel(s) were used	

APPENDIX 4

Additional question, remark on additional question and the comments

lab	description how the surface was measured and calculated
210	
339	
362	
551	First we divided the sample into six parts. Being three parts, isosceles triangles, a right triangle part, a square part, a rectangle part and the circular crown. Soon after, we measured the respective areas and summed them up. Only the golden contact area was considered, resulting in 131.27 mm2. When considered the golden area, plus the red contact area and the sides, the total area is 189.8985mm2. Golden contact area: 58.625 mm2. Red contact area: 58.625 mm2. Rectangular golden side area: Σ11 parts = 72.6485 mm2
623	
2102	We used a 3D-scanner
2108	The surface urea was divided in triangles and squares. This partial areas and the surface at the side were added up.
2115	the surface area was calculated with caliber
2121	Method A : Theoretical division into 6 triangles + the loop and measurement with a calliper. Method B : Image reprocessing software was used. The sample was photographed next to a calibrated ruler, in order to be able to a scale and measure certain data via said software.
2132	Measure the surface area by a calibrated digital caliper.
2138	Using by vernier callipers
2139	I divided each section of the sample and measured it by caliper.
2159	It was calculated by the sequeres method by taking the projection of the sample on the graph paper.
2165	Only calculate the golden part of the pendant. The golden part consist of a long edge, a ring, and a lightning pattern. The lightning pattern was divided into a rectangle, triangle, and trapezoid. Calculate their surface areas and add them all.
2184	
2201	The tested sample was divided to several approximate geometric form, total surface area is sum of surface area annuls, triangle, rectangle, trapezoid and sample perimeter, excluding the coated surface area
2213	We have considered the sample into two parts. One is triangular and another is cylindrical and accordingly we have measured the surface area of sample. We have calculated the surface area without substracting coated part (red coated part). Also please find the result after substracting the red coated part. After Substracting the coated part-171 mm2
2215	It was calculated by the sum of the parts: 71.08+54.74+21.90+42.47+22.60+212.79 (all in mm2)=2.13cm2
2216	Samples are sprayed with a whitener and scanned via 3D scanner. The scan is uploaded to computer software that calculates the total surface area.
2238	
2256	The total area is equal to the area of the ring plus the area of the pendant, the area of the pendant is equal to the bottom area plus the area all round.
2289	The red area could not be calculated. The sample can be divided into a ring and several squares. Calculate the area of the ring and the squares seperately, and then add this to obtain the final sample area
2290	
2293	Back side was divided in triangles and diamonds. Perimeter was calculated with thickness and length. Area of ring on top was calculated by taking the area of the circle within a circle and ring thickness and perimeter. All these areas were added for the final result. Area with enamel was not taken into account as for the nickel release assessment only areas in direct contact with the skin are evaluated.
2295	Total surface area: 244 mm2 Surface area without coated: 184 mm2
2300	Area of flat surface is measured with graph paper. Area of side surfaces and ring portion is measured with calibrated Vernier caliper.
2301	area divided into seven area, and some all to be as total surface area
2310	We calculate the surface area using by vernier caliper
2311	
2330	Use digital caliper for measured
2347	
2350	It was calculated by the sum of each part.
2352	
2357	
2363	
2365	
2366	
2369	
2370	This sample was measured and calculated via divided into 3 parts, including two planes as several triangle planes, circle part as a cylinder and sides as several rectangles.
2373	The red non-metallic part is not included in the total surface area
2375	
2377	The surface area was calibrated as the combination of different regular shape such as rectangle, triangle.
2378	
2379	By Vernier caliper
2380	This system consider as 2 triangle & 3 rectangle. Along with ring which as a rectangle. the both side of the items consider as a rectangle. the formula triangle=S$\sqrt{((S-a)(S)-b)(S-c)}$. Where a, b, c are the side of the triangle. The formula of the rectangle=axb. Where a & b are the side of the rectangle.
2381	In the pendant we found one isosceles triangle, Two rectangular, one quadrilateral & one circle.Area of one isosceles triangle is 37.053 mm2, Area of two rectangular is 167.256 mm2, Area of one quadrilateral is 14.64 mm2,Area of the circle is 13.645 mm2. Finally all the areas are added.
2382	A=5.06*4.54*2+(3.32*2.09/2)*2+2.58*6.21*2+((3.08+8.11)*2.5)/2)*2+40.75*1.55+1.33*13.23*2+1.13*13.32+2.28*1.13*2= 234 mm2

lab	description how the surface was measured and calculated
2385	The red part was excluded. The length and the width of the frame was measured by measuring each lenght from edge to edge. The bottom side was calculated by making two rectangular areas from the flash. The frame was included because it is of the same material as the bottom part and would be included in routine analysis. The frame was included for the calculation of the surface area. The area of the frame is 85 mm².
2390	Lower portion of flash shape pendant has taken as triangle and Mid portion is taken as rectangle. Upper portion of pendant divided into 3 triangle. sides have been taken as rectangle. At top of the pendant is circle. Surface area of these triangle, rectangle and circle.
2406	Caliper is used to determine the surface area of each sides, then total surface area are calculated.
2410	
2425	-
2426	Surface area calculated by vernier Caliper
2429	The area of the red part is not calculated, and the pattern is decomposed into several regular graphics, the surface area is calculated separately, and add the values together.
2442	Digital caliper was used to measured surface area. Calculation has been performed by using formula of rectangle and cylinder. Area calculation: Including plastic print - 237.06 mm2 Without plastic print - 178.06 mm2
2462	The sample is divided into regular shapes and then measured with a vernier caaliper
2475	
2485	Taken by a foto (smartphone), printed out, scanned as a pdf-file, the measurement of the surface was made with the "measurment tools" (polygon).
2489	Determination of area calculated based on the circle, rectangle, triangular formula.
2495	Use of a measuring software to determine the area 'A' of the lightning bolt and the length 'L' of its perimeter. Use of caliper to measure the thickness 't' of the object and the 2 radius of the torus and geometrical determination of its surface 'At=(2·pi·r·R)^2', so: Total Surfac Only one side of the lightning bolt was considered for the calculation because one side is enamelled.
2511	
2515	- Measurement apparatus: string and calliper - The sample is divided into different shapes to calculate the sample surface area: Trapezoid, rectangle, cylinder - Measure the sample area by marking the contour of the sample on graph paper - Measurement formulas use: Trapezoid, Rectangle, Cylinder
2532	Area calculated by using Vernier caliber and graph sheet.
2538	the area of the "flash" was (in mind) split into separate geometric forms like triangel and square; with a digital caliper the "separate" geometric forms were measured; the area of the ring was calculated by outer minus inner ring; minus the lost area of fixation at the flash; additional calulation: outer edge multiplied by the hight; outer and inner ring multiplied by the hight
2549	Sample area has been calculated by factorization of total surface area (Includes Rectanguler, Trianguler, Ring and Trapezium).
2560	Total surface area has been calculated considering different part as triangles, cylinder and side walls of pendant. 1. Total surface area including coated part is 240.02 mm2 2. Total surface area excluding coated part is 179.48 mm2
2573	
2582	The red colour area was divided into triangles and measured separately. Cylindric was measured separately
2590	I used the following geometrical figures: Cylinder, rectangles, triangles
2591	We measure the surface of the piece with a vernier caliper with a precision of 0.01mm.
2602	A digital measuring stick was used. The surface was subdivided in 3 areas: The eyelet (2 pi r * 2 pi r), the side face of the "lightning" (perimeter * thickness), the area of the "lightning" * 2. The area of the "lightning" (2x) was subdivided in geometric forms (triangles, polygon, trapeze).
2622	graph paper and caliber LB - 251 mm^2 is referred to sample #22576 + varnish - otherwise we found 200 mm^2 that is referred to sample #22576 without varnish.
2624	
2637	
2649	We considered the loop of the sample as a cylinder. For the front and back of body we used graph paper. For side we multiply the perimeter with depth.
2652	
2668	Circle + Triangle + Rectangle+ trapezium=Total surface area
2674	measured by Vernier caliper calculated by calculator
2678	The sample was seperated to triangles, rectangular shape measured by vernier caliper with 0.01 mm precision
2720	Red coated surface, entry area is not calculated.The sample is divided into two parts: ring and main body. The irregular main body is divided into several regular shapes and tested with vernier caliper. Finally, the areas of the two parts are added to obtain the final data
2721	A defined reference area paper together with the area of the trailer that comes into contact with the skin enlarged. Cut-out surfaces weighed. Only the area that comes into contact with the skin was calculated. Without sides and without front of the pendant.The longitudinal grooves were not taken into account.
2727	Use caliper. Aproximation to geometrical figures.
2737	Divide the sample into approximately regular parts, calculate the surface area of each part respectively, and add them to form the total surface area; The surface area of the coated part is not calculated.
2741	divided sample into 04 parts
2743	The total surface area was measured manually trying to split the sample in regular geometric shapes, getting the total surface by adding each part.
2758	We do not take in consideration the red surface
2818	S1=0.806 cm2; S2=0.756cm2; S3=0.588cm2; S1+S2+S3=2.150cm2
2826	The sample was divided into 1 triangle, 3 trapezoid and 1 ring. Sum up the area of 2 side of each of the triangle, trapezoid, the area of ring and the area of circumference.
2827	circle + Rectangle + Triangle + Rectangle + Triangle=Total surface area

lab	description how the surface was measured and calculated
2829	
2858	We consider sample as rectangular prism and solid cylinder. Surface area including red coating : 240.1 mm². Surface area excluding red coating : 182.5 mm².
2862	The pendant has been divided into many different geometric shapes. 1) Back pendant without ring suspension: Irregular square,10mm2, in the middle two trapezoids, 26mm2 and 18mm2, the apex a triangle, 7.5mm2, in Total 61,5mm2. 2) Outer Edge The rim 1.6 mm high, total 61mm2 3) Outline of the red coating 12mm2 4)outer edge ring 18mm2 5)flat ring hangers at the top and bottom 22mm
2864	Draw on a 10*10 mm2 drawing, calculate the grid.
2959	
3100	Divide the sample into solid rings, the front and the side. Then add the areas of the 3 parts.
3110	
3116	The area of each part were calculated by assuming their respective common geometrical shapes and each dimension was measured by using caliper.
3118	we divide the area into 4 parts on the gold surface, then calculated in part with a geometric approach. The sides are calculated according to the rectangle overall result in sum as total surface area
3146	Common geometric shapes were used to calculate the surface. Lightning: Calculated as a cuboid. The side surfaces of the lightning bolt were adjusted to the basic shape of the cuboid in order to fill the gaps. (1.712 mm²) Eyelet: Calculated as a washer. (0.550 mm²) Total area: 226.2 mm² for information only: verification of the area by using millimetre paper (214 mm²)
3153	The Pendant is calculated by dividing into 6 parts, including 2 trapezoids, 1 rectangle, thickness, O-ring, and edge of front. The dimensions of 6 parts are measured by digital caliper. The surface area of different parts are calculated by different formula, and add together to get the answer.
3160	We have not taken into account the red surface, as it is not metallic.
3172	Surface without coating 185.58 mm2
3182	Using Equation 1/2 x W x H for 4 point. Using Equation 1/2 x (parallel side effect) x H for 5 point. Using Equation 2 x 3.14 x r x h for 1 point. Using Equation W x L for 2 point. The result of surface area 227.85 mm2 calculate by without enamel. The result of total sample surface is 267.79 mm2
3185	The sample shapes are cutted into hollow cylinders, triangles, parallelograms and trapezoids, and then calculated with the corresponding area calculation formula
3197	Surface area of metal parts was calculated using formula of a few squares and triangles. Surface area of plastic part was not considered in total area.
3200	=2*（3.21*6.03*（1/2）+（6.22+6.49）/2*3.25+（5.25+7.25）/2*3.73+（1.72+4.01）/2*3.86）+1.54*（9.11+3.42+4.23+3.21+4.11+5.24+2.32+3.50+2.51+5.70）+2*3.14*（4.54/2）*1.54
3209	
3210	
3214	Measure the length and width of the metal part, plus the surface area of the cut surface.
3218	Total surface area=Ring + gold back + side
3228	The sample is divided into a ring, a triangle, three trapezoids. Calculate the perimeter and multiply the perimeter by the thickness to get the area of the side. Add up these areas.
3230	Area of electric shape:14.42 + 18.35 + 21.47 + 15.74 = 69.979 mm2; Area of ring: (2 x π x (2.305x2.305) + 2 x π x 2.305 x 1.14) – (2 x π x 1.345 x 1.14) = 40.259 mm2; Area of edges: (7.967 + 2.82 + 4.3068 + 8.0184 + 13.9832 + 3.5372 + 5.1636 + 3.8808 + 5.4912 + 6.851) mm2 = 62.0192 mm Area of pendant = 69.979 + 40.259 + 62.0192 = 172.257 mm2 Measurement was done using a vernier calliper
3246	Assume it into 5 triangles and 2 Parallelograms, and 1 rectangle

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
7 labs in GERMANY
1 lab in GUATEMALA
8 labs in HONG KONG
9 labs in INDIA
3 labs in INDONESIA
8 labs in ITALY
4 labs in KOREA, Republic of
1 lab in MAURITIUS
1 lab in MOROCCO
31 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in PORTUGAL
2 labs in SPAIN
1 lab in SRI LANKA
3 labs in SWITZERLAND
3 labs in TAIWAN
2 labs in THAILAND
1 lab in THE NETHERLANDS
2 labs in TUNISIA
4 labs in TURKEY
1 lab in U.S.A.
4 labs in VIETNAM

APPENDIX 6

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01), D1	= outlier in Dixon's outlier test
D(0.05), D5	= straggler in Dixon's outlier test
G(0.01),G1	= outlier in Grubbs' outlier test
G(0.05),G5	= straggler in Grubbs' outlier test
DG(0.01),DG1	= outlier in Double Grubbs' outlier test
DG(0.05), DG5	= straggler in Double Grubbs' outlier test
R(0.01), R1	= outlier in Rosner's outlier test
R(0.05), R5	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from the statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported

Literature

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
8 J.N. Miller, Analyst, 118, 455, (1993)
9 Analytical Methods Committee, Technical Brief, No 4, January 2001
10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)