Institute for
Interlaboratory Studies

Results of Proficiency Test
Nickel release and
Surface determination
June 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Nickel has always been used in various applications for example as a plated substance on another metal or as an alloy. Nickel applications usually do not give problems, but when Nickel comes to 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. 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. Nickel containing items that are used in prolonged human contact are tested for Nickel release. This to avoid products on the market with too much Nickel release. With this regulation it prevents that more people become sensitized. Products, like jewelry in piercings (earrings), watches or clothes fasteners, are tested in compliance with entry No. 27 to Annex XVII of Regulation (CE) 1907/2006 (REACH).

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 2022/2023 it was decided to continue the proficiency test for the determination of Nickel release and Surface Determination.

In this interlaboratory study 101 laboratories in 27 countries registered for participation, see appendix 5 for the number of participants per country. In this report 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 #23625 and one piece of a pendant labelled #23626 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 purchased from a local retailer. 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. In total 130 subsamples of three pieces each were prepared and labelled #23625.
The homogeneity of the subsamples was checked by determination of Nickel release using test method EN1811 on eight stratified randomly selected subsamples. Please note, a subsample is one bag with three items.

	Nickel release in µg/cm^2/week
sample #23625-1	0.425
sample #23625-2	0.483
sample #23625-3	0.575
sample #23625-4	0.474
sample #23625-5	0.525
sample #23625-6	0.458
sample #23625-7	0.500
sample #23625-8	0.517

Table 1: homogeneity test results of subsamples #23625

From the above test results the relative standard deviation (RSD) was calculated and compared with 0.3 times the relative standard deviation estimated from the average of PT uncertainties of previous PTs from 2014-2021 in agreement with the procedure of ISO13528, Annex B2, in the next table.

	Nickel release
RSD (observed)	9.2%
reference method	iis PTs
0.3 x RSD (reference method)	8.5%

Table 2: evaluation of the relative standard deviation of subsamples #23625

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

For the determination of surface determination only a batch of metal leaves, which were parts from earrings, was purchased from a local retailer. From this batch 130 small plastic bags were filled with one leaf. This small bag was placed in an outer bag and labelled #23626. No homogeneity tests were done over the subsamples because only surface determination has been requested for this sample. However, each leaf was weighed in advance to make sure that no large differences exist in the surfaces.

To each of the participating laboratories one sample #23625 and one sample #23626 were sent on June 7, 2023.

2.5 ANALYZES

The participants were requested to determine Nickel release on sample #23625 and to determine the surface only on sample #23626.
The participants were informed that the metal plates of sample #23625 are non-coated and a simulation of accelerated wear and corrosion did not need to be conducted. Also, that on critical examination some small irregularities such as slight scratches on the surface of the sample may be observed. The influence of these irregularities on the nickel release is negligible. This was proven by testing prior to use in this PT.
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:

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

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. Nine participants reported test results after the final reporting date and four other participants did not report any test results.
In total 97 participants reported 289 test results numerical test results. Observed were 9 outlying test results which is 3.1%. 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 clear precision statement that mentions a repeatability and/or a reproducibility. In Annex A of test method EN1811:11+A1:15 is mentioned that the measurement uncertainty in a 2008 interlaboratory study was 46%, while in Annex B is stated that “The relative test method reproducibility in this ILC was 33.3%”. As it is not clear which of the two statements, both mentioned in annexes, should be used for the target reproducibility it was decided to use a target reproducibility calculated with the Horwitz equation. This target obtained from Horwitz 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.

Please note, a new version of test method EN1811has been published in August of 2023.

sample #23625

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

Surface determination: The determination on the square piece may not be problematic. Six statistical outliers were observed in the reported range of 9.01-10.0 cm^2. No official test method exists for Surface determination. Therefore, no z-scores were calculated. The relative standard deviation for this sample after rejection of the statistical outliers is 1.0%.

sample #23626

Surface determination: This determination on a leaf may be problematic. Two statistical outliers were observed in the reported range of 0.33-1.13 cm^2. No official test method exists for Surface determination. Therefore, no z-scores were calculated. The relative standard deviation for this sample is 19% and larger than the range of the observed relative standard deviations in previous iis PTs (3.5-13%). It is also larger compared to the relative standard deviation of the Surface determination of the simpler shaped sample #23625 (1.0%). 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	µg/cm^2/week	96	0.51	0.32	0.25
Surface determination	cm^2	91	9.58	0.26	n.a.

Table 3: reproducibility of tests on sample #23625

Without further statistical calculations it can be concluded that for the determination of Nickel release there is not a good compliance of the group of participants with the target reproducibility.

Parameter	unit	n	average	2.8 * sd	R(target)
Surface determination	cm^2	93	0.86	0.47	n.a.

Table 4: reproducibility of tests on sample #23626

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2023 WITH PREVIOUS PTS

	June 2023	April 2022	June 2021	June 2020	June 2019
Number of reporting laboratories	97	112	96	104	127
Number of test results	289	331	191	205	126
Number of statistical outliers	9	11	7	11	5
Percentage statistical outliers	3.1	3.3%	3.7%	5.4%	4.0%

Table 5: 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	June 2023	April 2022	June 2021	June 2020	2019-2014
Nickel release	22%	15%	21%	29%	18 - 44%
Surface determination	1.0-19%	0.9 – 13%	0.7 – 6.9%	2.5 – 3.5%	1.1 - 13%

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

The uncertainty of the determination of Nickel release sample #23625 is larger in comparison with the uncertainty from last year and in line with previous years.
The uncertainty of the Surface determination of sample #23626 was larger when compared to previous PTs. This is probably due to the complex shape of the sample.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For the Nickel release sample #23625 the following can be summarized from the analytical details as reported by the participants. The details are given in appendix 2 and 3:

- 87% of the participants mentioned to be accredited for the determination of Nickel release.
- 63% of the participants have used new or disposable test vessels, 36 % used previously used test vessels. Of these previously used test vessels 27 participants has done a pretreatment and remarkably 4 participants have not done any pretreatment.
- Around 70% of the reporting participants used a ratio of approximately 1 mL test solution per cm^2 sample surface area. 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.

For sample #23626 a variety of methods for the surface determination was described by 63% of the reporting participants. See appendix 4 for these detailed descriptions.

5 DISCUSSION

The calculated in-between item repeatability on the Nickel release results between the three items of sample #23625 showed a large variation over the participants (see appendix 2). The RSDr varies from 0.9% to 40.7%. One participant reported an extremely large RSDr of 109.1%. Only 33 participants reported test results with a RSDr in agreement with the target repeatability standard deviation of 4%. This 4% is estimated from EN1811:11+A1:15 as follows: 33.3% / 2.8 / 3. When evaluating the test results of only those 33 participants the average is not significantly different compared to the whole group of 96 participants. Remarkably, the variation over the test results of this subgroup is better and is in agreement with the estimated reproducibility calculated with the Horwitz equation. See appendix 1 for the summary of this sub evaluation.

6 CONCLUSION

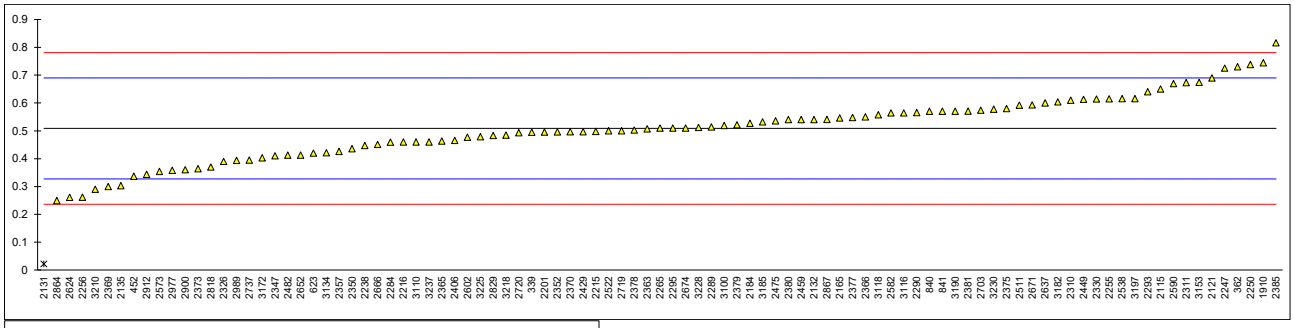
It can be concluded that the majority of the participants had no problems with the determination of Nickel release.
However, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and increase the quality of the analytical results.

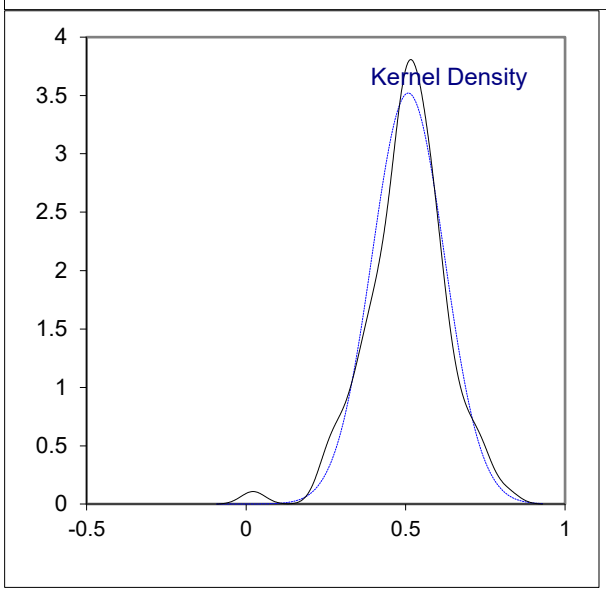
APPENDIX 1

Determination of Nickel release on sample #23625; average result of three replicates in µg/cm²/week

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
339	EN1811	0.4953		-0.15	
362		0.73		2.44	
452		0.3366		-1.90	
551		-----		-----	
623	EN1811	0.42		-0.98	
840	EN1811	0.57		0.68	
841	EN1811	0.57		0.68	
1910	EN1811 + A1	0.7445		2.60	
2115	EN1811	0.65		1.56	
2121	EN1811 + A1	0.69		2.00	
2131	EN1811	0.02205	R(0.01)	-5.37	
2132	EN1811 + A1	0.5405		0.35	
2135	EN1811	0.303		-2.27	
2165	EN1811 + A1	0.546		0.41	
2184	EN1811	0.527		0.20	
2201	EN1811 + A1	0.4958		-0.14	
2215	EN1811	0.4980		-0.12	
2216	EN1811	0.46		-0.54	
2236		-----		-----	
2238	EN1811 + A1	0.447		-0.68	
2247	EN1811	0.725		2.39	
2250	EN1811	0.7384		2.53	
2255	EN1811 + A1	0.615		1.17	
2256	EN1811	0.262		-2.72	
2265	EN1811	0.51		0.01	
2284	EN1811	0.459		-0.55	
2289	EN1811 + A1	0.514		0.06	
2290	EN1811	0.566		0.63	
2293	EN1811	0.6401		1.45	
2295	EN1811 + A1	0.51		0.01	
2310	EN1811	0.61		1.12	
2311	EN1811	0.673		1.81	
2326	EN1811	0.390	C	-1.31	First reported 0.882
2330	EN1811 + A1	0.614		1.16	
2347	EN1811 + A1	0.41		-1.09	
2350	EN1811 + A1	0.436		-0.80	
2352	EN1811	0.496		-0.14	
2357	EN1811	0.426		-0.91	
2363	EN1811 + A1	0.507		-0.02	
2365	EN1811 + A1	0.4634		-0.50	
2366	EN1811	0.55		0.46	
2369	EN1811	0.300		-2.30	
2370	EN1811	0.497		-0.13	
2373	EN1811	0.364		-1.60	
2375	EN1811	0.58		0.79	
2377	EN1811	0.548		0.43	
2378	EN1811	0.503		-0.06	
2379	EN1811	0.522		0.15	
2380	EN1811 + A1	0.540		0.35	
2381	EN1811	0.571		0.69	
2385	EN1811	0.816		3.39	
2406	EN1811	0.4659		-0.47	
2429	EN1811	0.497		-0.13	
2449	EN1811	0.6133		1.15	
2459	EN1811	0.54		0.35	
2475	EN1811	0.536		0.30	
2482	EN1811	0.4129		-1.06	
2511	EN1811	0.592		0.92	
2522	EN1811	0.50		-0.10	
2538	EN1811	0.616		1.18	
2573	EN1811	0.354		-1.71	
2582	EN1811	0.564		0.61	
2590	EN1811	0.670	C	1.78	First reported 0.22
2602	EN1811	0.477		-0.35	
2624	EN1811 + A1	0.261		-2.73	
2637	EN1811	0.6		1.01	
2652	EN1811	0.413		-1.05	
2666	EN1811 + A1	0.451		-0.64	
2671	EN1811	0.5933		0.93	
2674	EN1811	0.51		0.01	
2678		-----		-----	
2703	EN1811 + A1	0.574		0.72	
2719	EN1811	0.50		-0.10	
2720	EN1811	0.4936		-0.17	

lab	method	value	mark	z(targ)	remarks
2737	EN1811	0.395		-1.25	
2818	EN1811	0.370		-1.53	
2829	EN1811 + A1	0.483		-0.28	
2864	EN1811 + A1	0.25		-2.85	
2867	EN1811	0.541		0.36	
2900		0.360		-1.64	
2912	EN1811 + A1	0.344		-1.82	
2977	EN1811	0.358	C	-1.66	First reported 0.217
2989	EN1811	0.3940		-1.26	
3100	EN1811	0.519		0.11	
3110	EN1811 + A1	0.46		-0.54	
3116	EN1811 + A1	0.564		0.61	
3118		0.5576		0.54	
3134	EN1811 + A1	0.421187		-0.96	
3153	EN1811 + A1	0.674		1.82	
3172	EN1811	0.403		-1.17	
3182	EN1811	0.604		1.05	
3185	EN1811 + A1	0.532		0.26	
3190	EN1811	0.57		0.68	
3197	EN1811	0.616		1.18	
3210	EN1811	0.29		-2.41	
3218	EN1811	0.485		-0.26	
3225	EN1811 + AC	0.4787		-0.33	
3228	EN1811	0.512		0.04	
3230	In house	0.5776	C	0.76	First reported 0.7907
3237	EN1811 + A1	0.46		-0.54	
					Only RSD <4% between replicates
	normality	OK			OK
	n	96			33
	outliers	1			0
	mean (n)	0.509			0.504
	st.dev. (n)	0.1133	RSD = 22%		0.0909 RSD = 18%
	R(calc.)	0.317			0.255
	st.dev.(Horwitz)	0.0907			0.0899
	R(Horwitz)	0.254			0.252
compare					
	R(EN1811:11+A1:15)	0.169			0.168



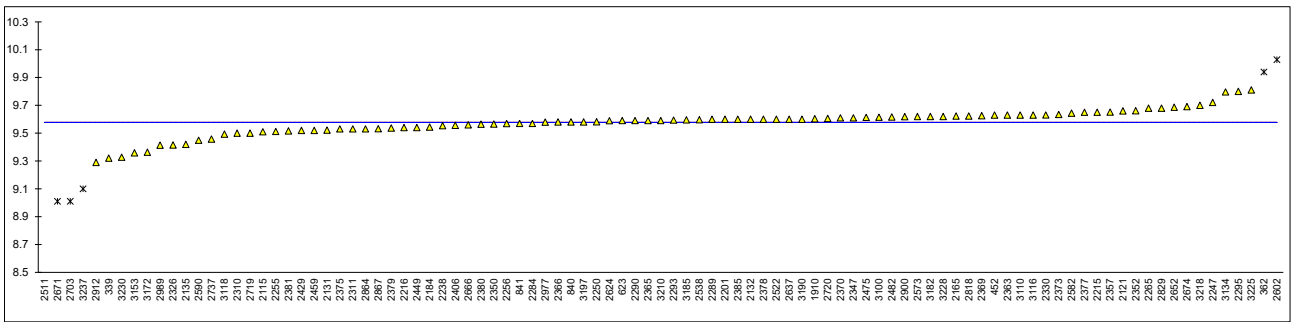


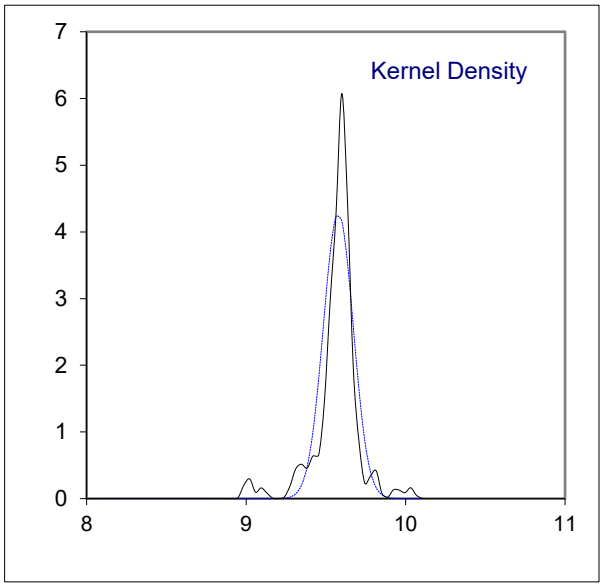
Determination of Surface on sample #23625; results in cm^2

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
339		9.32		-----	
362		9.94	R(0.01)	-----	
452		9.63		-----	
551		-----		-----	
623		9.59		-----	
840		9.58		-----	
841		9.57		-----	
1910		9.603		-----	
2115		9.51		-----	
2121		9.6598		-----	
2131		9.522		-----	
2132		9.600		-----	
2135		9.42		-----	
2165		9.623		-----	
2184		9.544		-----	
2201		9.60		-----	
2215		9.65		-----	
2216		9.54		-----	
2236		-----		-----	
2238		9.554		-----	
2247		9.721		-----	
2250		9.582		-----	
2255		9.512		-----	
2256		9.569		-----	
2265		9.68		-----	
2284		9.57		-----	
2289		9.60		-----	
2290		9.59		-----	
2293		9.594		-----	
2295		9.8		-----	
2310		9.5		-----	
2311		9.53		-----	
2326		9.415		-----	
2330		9.631		-----	
2347		9.61		-----	
2350		9.565		-----	
2352		9.661		-----	
2357		9.651		-----	
2363		9.63		-----	
2365		9.59		-----	
2366		9.58		-----	
2369		9.625		-----	
2370		9.61		-----	
2373		9.634		-----	
2375		9.53		-----	
2377		9.65		-----	
2378		9.60		-----	
2379		9.537		-----	
2380		9.564		-----	
2381		9.516		-----	
2385		9.6		-----	
2406		9.5564		-----	
2429		9.520		-----	
2449		9.54		-----	
2459		9.52		-----	
2475		9.613		-----	
2482		9.616		-----	
2511		0.98	R(0.01)	-----	
2522		9.60		-----	
2538		9.597		-----	
2573		9.62		-----	
2582		9.6438		-----	
2590		9.449		-----	
2602		10.028	R(0.01)	-----	
2624		9.589		-----	
2637		9.6		-----	
2652		9.686		-----	
2666		9.56		-----	
2671		9.01	R(0.01)	-----	
2674		9.69		-----	
2678		-----		-----	
2703		9.01	R(0.01)	-----	
2719		9.5		-----	
2720		9.606		-----	

lab	method	value	mark	z(targ)	remarks
2737		9.458		-----	
2818		9.623		-----	
2829		9.68		-----	
2864		9.53		-----	
2867		9.532		-----	
2900		9.6185		-----	
2912		9.29		-----	
2977		9.5794		-----	
2989		9.4140		-----	
3100		9.614		-----	
3110		9.63		-----	
3116		9.63		-----	
3118		9.4935		-----	
3134		9.7958		-----	
3153		9.359		-----	
3172		9.363		-----	
3182		9.62		-----	
3185		9.596		-----	
3190		9.6		-----	
3197		9.58		-----	
3210		9.59		-----	
3218		9.70		-----	
3225		9.81		-----	
3228		9.62		-----	
3230		9.327		-----	
3237		9.1	R(0.01)	-----	

	normality	suspect
	n	91
	outliers	6
	mean (n)	9.577
	st.dev. (n)	0.0932 RSD = 1.0%
	R(calc.)	0.261



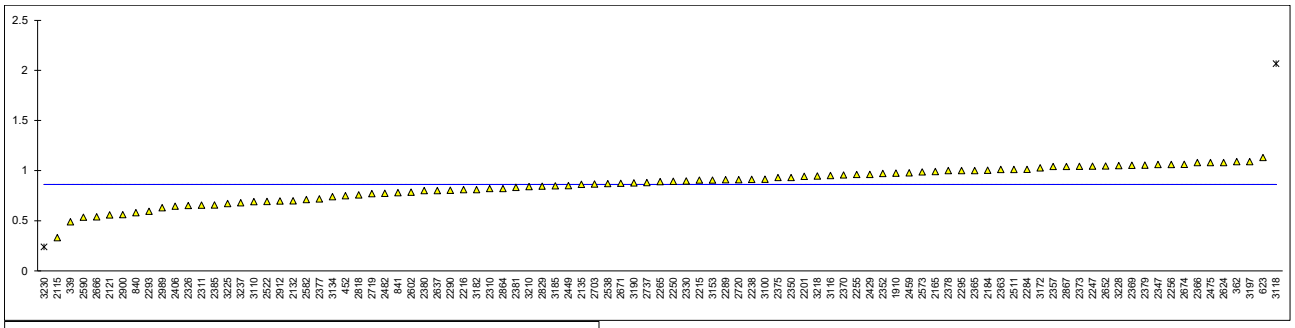


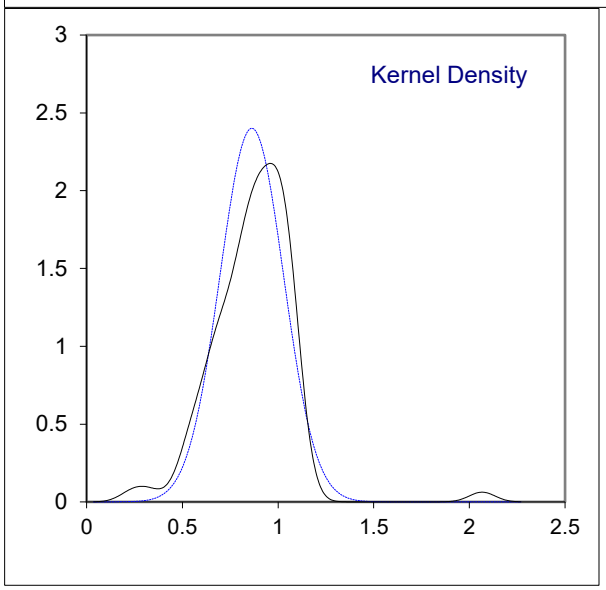
Determination of Surface on sample #23626; results in cm^2

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
339		0.49		-----	
362		1.09		-----	
452		0.75		-----	
551		-----		-----	
623		1.13		-----	
840		0.58		-----	
841		0.78		-----	
1910		0.975		-----	
2115		0.33		-----	
2121		0.5579		-----	
2131		-----		-----	
2132		0.6977		-----	
2135		0.8622		-----	
2165		0.991		-----	
2184		1.003		-----	
2201		0.942		-----	
2215		0.9050		-----	
2216		0.81		-----	
2236		-----		-----	
2238		0.913		-----	
2247		1.044		-----	
2250		0.8927		-----	
2255		0.961		-----	
2256		1.06009		-----	
2265		0.89		-----	
2284		1.012		-----	
2289		0.91		-----	
2290		0.804		-----	
2293		0.5930		-----	
2295		1.00		-----	
2310		0.82		-----	
2311		0.655		-----	
2326		0.651		-----	
2330		0.8953		-----	
2347		1.06		-----	
2350		0.931		-----	
2352		0.972		-----	
2357		1.041		-----	
2363		1.01		-----	
2365		1.00		-----	
2366		1.08		-----	
2369		1.053		-----	
2370		0.956		-----	
2373		1.043		-----	
2375		0.93		-----	
2377		0.718		-----	
2378		1.0		-----	
2379		1.054		-----	
2380		0.80		-----	
2381		0.832		-----	
2385		0.657		-----	
2406		0.6453		-----	
2429		0.963		-----	
2449		0.85		-----	
2459		0.977		-----	
2475		1.08		-----	
2482		0.7742		-----	
2511		1.01		-----	
2522		0.692		-----	
2538		0.870		-----	
2573		0.986		-----	
2582		0.712		-----	
2590		0.534		-----	
2602		0.784		-----	
2624		1.08		-----	
2637		0.8		-----	
2652		1.045		-----	
2666		0.540		-----	
2671		0.873		-----	
2674		1.062		-----	
2678		-----		-----	
2703		0.86583924		-----	
2719		0.77		-----	
2720		0.91		-----	

lab	method	value	mark	z(targ)	remarks
2737		0.8803		-----	
2818		0.756	C	-----	Reported: 75.617 cm²
2829		0.845		-----	
2864		0.82		-----	
2867		1.041		-----	
2900		0.56		-----	
2912		0.697		-----	
2977		-----		-----	
2989		0.63		-----	
3100		0.914		-----	
3110		0.69		-----	
3116		0.95		-----	
3118		2.0664	R(0.01)	-----	
3134		0.74		-----	
3153		0.905		-----	
3172		1.028		-----	
3182		0.81		-----	
3185		0.848		-----	
3190		0.8781		-----	
3197		1.09		-----	
3210		0.84		-----	
3218		0.946		-----	
3225		0.67		-----	
3228		1.05		-----	
3230		0.23904	R(0.05)	-----	
3237		0.68		-----	

normality	OK
n	93
outliers	2
mean (n)	0.863
st.dev. (n)	0.1662
R(calc.)	0.465
	RSD = 19%





APPENDIX 2

Determination of Nickel release and some Analytical Details on subsamples #23625

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	-----	-----	-----	-----		
339	0.4505	0.513	0.5225	7.9	9.3	1/1
362	0.726	0.799	0.677	8.4		
452	0.3505	0.3219	0.3375	4.3	10	1 ml/cm2
551	-----	-----	-----	-----		
623	0.48	0.39	0.40	11.7	20	1:1
840	0.52	0.63	0.57	9.7	9.6L	1:1
841	0.52	0.63	0.57	9.7	10	1:1
1910	0.6855	0.7882	0.7598	7.1	35	3,6:1
2115	0.53	0.66	0.75	17.0	12	
2121	0.57	0.58	0.75	14.7	20	
2131	0.02312	0.02099	0.02206	4.8	20	2.1
2132	0.6124	0.5475	0.4616	14.0	10	1 : 0.96
2135	0.372	0.377	0.161	40.7	15	1:1,59
2165	0.547	0.577	0.514	5.8	10	10ml:9.62cm²
2184	0.532	0.545	0.503	4.1	10	1:1
2201	0.5152	0.4894	0.4829	3.4	9.6	1:1
2215	0.5050	0.5004	0.4887	1.7	13	13:9.65
2216	0.37	0.50	0.51	17.0	10	~1:1
2236	-----	-----	-----	-----		
2238	0.434	0.449	0.459	2.8	10	1.05
2247	0.768	0.711	0.695	5.3	10	1:1
2250	0.8462	0.6134	0.7556	15.9	12	1,252
2255	0.587	0.618	0.639	4.3	10	1:1
2256	0.326	0.219	0.240	21.6	20	2.09
2265	0.50	0.56	0.46	9.9	20,0	2/1
2284	0.432	0.462	0.482	5.5	9.6	1:1
2289	0.507	0.523	0.512	1.6	9.6	1:1
2290	0.527	0.577	0.593	6.1		
2293	1.4462	0.2502	0.2241	109.1	25	2.6
2295	0.50	0.52	0.52	2.3	12	1.22
2310	0.61	0.64	0.57	5.8	10	1:1
2311	0.667	0.682	0.671	1.2	9.55	1:1
2326	0.387	0.382	0.403	2.8	9.4	9.4 ml / 9.145 cm2
2330	0.593	0.719	0.531	15.6	10	1:1
2347	0.411	0.408	0.421	1.7	9.61	1:1
2350	0.493	0.392	0.423	11.9	10	10/9.57= 1.04 mL/cm2
2352	0.494	0.507	0.488	2.0	15	2:1
2357	0.431	0.415	0.434	2.4		
2363	0.525	0.442	0.553	11.4	9.63	1:1
2365	0.4356	0.4717	0.4829	5.3	10l	1:1
2366	0.54	0.55	0.55	1.0		
2369	0.298	0.310	0.292	3.1		
2370	0.524	0.481	0.486	4.7	10	1:1
2373	0.361	0.382	0.349	4.6	9.63	1:1
2375	0.59	0.52	0.61	8.1	10	1:1
2377	0.562	0.553	0.530	3.0	9.7	1
2378	0.49	0.52	0.50	3.0	9.6	1mL/cm²
2379	0.499	0.542	0.523	4.1	9.60	1:1
2380	0.561	0.487	0.572	8.6	12	1:1
2381	0.571	0.592	0.551	3.6	10	1:1
2385	0.735	0.846	0.866	8.6		
2406	0.4380	0.4446	0.5152	9.2	9.6	1mL:1cm2
2429	0.543	0.449	0.498	9.5	9.6	1:1
2449	0.61	0.61	0.62	0.9	10	1:1
2459	0.49	0.54	0.56	6.7	15	1.57
2475	0.586	0.572	0.451	13.8	9.60	1
2482	0.4126	0.3782	0.4479	8.4	9	1:1
2511	0.595	0.607	0.575	2.7		
2522	0.51	0.53	0.47	6.1	50	5
2538	0.697	0.593	0.558	11.7	10	1 ml : 1 cm2
2573	0.370	0.355	0.338	4.5	9.6	1:1
2582	0.495	0.580	0.619	11.2	10	1:1
2590	0.681	0.640	0.691	4.0	20	2 mL/cm²
2602	0.444	0.479	0.515	7.4	10	1:1
2624	0.239	0.333	0.414	33.6	15	1:1,5
2637	0.57	0.62	0.59	4.2		
2652	0.442	0.393	0.405	6.2		
2666	0.455	0.447	0.450	0.9	15	1.57
2671	0.62	0.59	0.57	4.2	10	1:1
2674	0.492	0.498	0.526	3.6	11	1.14
2678	-----	-----	-----	-----		
2703	0.526	0.535	0.662	13.2	16	1.7:1
2719	0.44	0.50	0.55	11.0		

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)
2720	0.4875	0.5031	0.4901	1.7	9.61	1:1
2737	0.428	0.387	0.369	7.7	15	1.58 mL/cm²
2818	0.411	0.370	0.329	11.1		
2829	0.427	0.424	0.597	20.5	11	1.14
2864	0.26	0.23	0.26	6.9	10	1:1
2867	0.544	0.573	0.506	6.2	9.5	1 : 1
2900	0.335	0.428	0.317	16.5		
2912	0.355	0.335	0.343	2.9	20	2.15 mL/cm²
2977	0.425	0.333	0.317	16.3	10	about 1
2989	0.3920	0.3840	0.4040	2.6	9.41	1:1
3100	0.512	0.496	0.548	5.1	9.6	1:1
3110	0.46	0.45	0.48	3.3		
3116	0.563	0.604	0.526	6.9	10	1
3118	0.6068	0.5142	0.5518	8.4	9	1:1
3134	0.40745	0.44713	0.40898	5.3	10	1:1
3153	0.766	0.579	0.678	13.9	9.4	1:1
3172	0.41	0.43	0.37	7.6		
3182	0.616	0.625	0.571	4.8	9.62	1:1
3185	0.492	0.593	0.511	10.1	10.0	10.0:9.60:0.53
3190	0.57	0.50	0.65	13.2	9.6	1:1
3197	0.570	0.597	0.680	9.3	12.1	1.26:1
3210	0.32	0.24	0.32	15.9	20	2.09
3218	0.406	0.494	0.555	15.4	9.7	1:1
3225	0.4815	0.4717	0.4829	1.3	9.8	1ml to 1cm2
3228	0.520	0.509	0.507	1.4	9.62	1
3230	0.572	0.583	-----	1.3	15	1:1.61
3237	0.51	0.43	0.43	10.0	15	1,64

APPENDIX 3

Other reported Analytical Details for sample #23625

lab	ISO17025 accred.	pre-treat test vessel	pre-treatment procedure
210	---	---	
339	No	No, new/disposable test vessel(s) were used	
362	Yes	No, new/disposable test vessel(s) were used	
452	No	No, new/disposable test vessel(s) were used	
551	---	---	
623	Yes	Other, please specify in the remarks below	
840	Yes	Yes, the previously used test vessel(s) were pre-treated	
841	Yes	Yes, the previously used test vessel(s) were pre-treated	Ultrasonicate the test flask with Hno3 % for at least 30 minutes
1910	No	Yes, the previously used test vessel(s) were pre-treated	The previously used vessels were pre-treated with 5% nitric acid for 4 hours
2115	Yes	No, the previously used test vessel(s) were not pre-treated	
2121	Yes	No, new/disposable test vessel(s) were used	
2131	Yes	No, the previously used test vessel(s) were not pre-treated	
2132	Yes	No, new/disposable test vessel(s) were used	
2135	Yes	No, new/disposable test vessel(s) were used	
2165	Yes	No, new/disposable test vessel(s) were used	
2184	Yes	No, new/disposable test vessel(s) were used	
2201	Yes	Yes, the previously used test vessel(s) were pre-treated	Nitric acid dilute 5%,cleaning 4 hours
2215	Yes	Yes, the previously used test vessel(s) were pre-treated	Stored in 5% nitric acid for 4 H, rinse and holder with deionised water and dry.
2216	Yes	Yes, the previously used test vessel(s) were pre-treated	Soaked new test vessels in 5% HNO3 for 4 hours
2236	---	---	
2238	No	No, new/disposable test vessel(s) were used	
2247	Yes	No, new/disposable test vessel(s) were used	-
2250	Yes	No, new/disposable test vessel(s) were used	
2255	Yes	No, new/disposable test vessel(s) were used	NA
2256	Yes	Yes, the previously used test vessel(s) were pre-treated	pre-treated with diluted Nitric acid for overnight
2265	No	No, new/disposable test vessel(s) were used	
2284	Yes	No, new/disposable test vessel(s) were used	
2289	Yes	No, new/disposable test vessel(s) were used	
2290	Yes	---	
2293	Yes	No, the previously used test vessel(s) were not pre-treated	
2295	Yes	No, new/disposable test vessel(s) were used	
2310	Yes	No, new/disposable test vessel(s) were used	
2311	Yes	Yes, the previously used test vessel(s) were pre-treated	Pretreat with 5% Nitric acid, rinsed with deionized water and dried.
2326	Yes	No, new/disposable test vessel(s) were used	
2330	Yes	Yes, the previously used test vessel(s) were pre-treated	
2347	Yes	No, new/disposable test vessel(s) were used	/
2350	Yes	Yes, the previously used test vessel(s) were pre-treated	
2352	Yes	No, new/disposable test vessel(s) were used	
2357	---	---	
2363	Yes	Yes, the previously used test vessel(s) were pre-treated	use 5% HNO3 stay for 4 hours
2365	Yes	No, new/disposable test vessel(s) were used	
2366	Yes	---	
2369	---	---	
2370	Yes	No, new/disposable test vessel(s) were used	
2373	Yes	No, new/disposable test vessel(s) were used	
2375	Yes	No, new/disposable test vessel(s) were used	-
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 hrs
2380	Yes	Yes, the previously used test vessel(s) were pre-treated	All vessels shall be pre-treated by being stored in dilute nitric acid for at least 4 hours. Then rinse with deionized water & dry.
2381	Yes	Yes, the previously used test vessel(s) were pre-treated	All test vessels are pretreated by 5% Nitric acid for 4 hours and then rinsed with deionized water and dry.

lab	ISO17025 accred.	pre-treat test vessel	pre-treatment procedure
2385	Yes	No, new/disposable test vessel(s) were used	
2406	Yes	Yes, the previously used test vessel(s) were pre-treated	Rinse with tap water, then soak in 10% nitric acid bath for 2 hr, rinse with DI water then dry in oven overnight before use
2429	Yes	No, new/disposable test vessel(s) were used	
2449	No	No, new/disposable test vessel(s) were used	
2459	Yes	Yes, the previously used test vessel(s) were pre-treated	5% Nitric Acid for 4 hours
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
2482	Yes	No, new/disposable test vessel(s) were used	
2511	---	---	
2522	No	No, new/disposable test vessel(s) were used	
2538	Yes	No, new/disposable test vessel(s) were used	
2573	Yes	No, the previously used test vessel(s) were not pre-treated	
2582	Yes	No, new/disposable test vessel(s) were used	
2590	Yes	No, new/disposable test vessel(s) were used	
2602	Yes	No, new/disposable test vessel(s) were used	
2624	Yes	No, new/disposable test vessel(s) were used	
2637	No	---	
2652	Yes	Yes, the previously used test vessel(s) were pre-treated	
2666	Yes	No, new/disposable test vessel(s) were used	
2671	Yes	Yes, the previously used test vessel(s) were pre-treated	pre treated with nitric acid. Rinsed with deionized water & dried
2674	Yes	Yes, the previously used test vessel(s) were pre-treated	5%HNO3
2678	---	---	
2703	No	No, new/disposable test vessel(s) were used	
2719	Yes	Yes, the previously used test vessel(s) were pre-treated	
2720	Yes	Yes, the previously used test vessel(s) were pre-treated	Soak in 5% nitric acid for 4 hours
2737	Yes	No, new/disposable test vessel(s) were used	
2818	---	---	
2829	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	---	---	
2912	No	No, new/disposable test vessel(s) were used	
2977	Yes	No, new/disposable test vessel(s) were used	
2989	Yes	No, new/disposable test vessel(s) were used	
3100	Yes	Yes, the previously used test vessel(s) were pre-treated	Vessels with lids,the vessels shall be pre-treated by being stored in a solution of 5% dilute nitril acid for at least 4 hours.After acid treatment,rinse the vessel with Grade I water and dry.
3110	---	---	
3116	Yes	Yes, the previously used test vessel(s) were pre-treated	Pre-treated in a solution of diluted nitric acid (~5%) for at least 4 hours. Then rinse with deionized water and dry.
3118	Yes	No, new/disposable test vessel(s) were used	-
3134	No	No, new/disposable test vessel(s) were used	
3153	Yes	No, new/disposable test vessel(s) were used	
3172	Yes	---	
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	Immersion with 5% HNO3 nitric acid solution
3190	Yes	No, new/disposable test vessel(s) were used	/
3197	Yes	No, new/disposable test vessel(s) were used	
3210	Yes	No, new/disposable test vessel(s) were used	
3218	Yes	No, new/disposable test vessel(s) were used	
3225	Yes	No, new/disposable test vessel(s) were used	/
3228	Yes	No, new/disposable test vessel(s) were used	
3230	Yes	Yes, the previously used test vessel(s) were pre-treated	The plastic containers were pre-treated by storing in 5% nitric acid for at least 4 hours (Same as corrosion procedure)
3237	Yes	No, new/disposable test vessel(s) were used	

APPENDIX 4

Detailed description of the Surface Determination on sample #23626

lab	description how the surface was measured and calculated
210	
339	Using a calliper and a spreadsheet with common shapes.
362	
452	using geometrical shapes.
551	
623	
840	The leaf part is calculated according to the rhombus plus the o-ring part is calculated according to the circle, and minus the holes in the leaf.
841	We split the sample into thumbnails and then sum the thumbnails.
1910	The area was measured with a calliper. The ellipse area formula was used for the calculations.
2115	
2121	
2131	
2132	Measure the surface area with graph paper.
2135	with calliper, calculated as Ellipse, The size of the holes was estimated.
2165	
2184	
2201	This leaf is seen as a circle, an ellipse, and a triangle. Subtract the hollow area and add the side area.
2215	SA=21.93mm2 SB=39.4mm2 SC=24.68mm2 S16=8.84mm2 S1+S2+S3+S4+S5+S6+S7+S8+S9+S10+S11+S12+S13+S14+S15=4.35mm2 STOTAL=SA+SB+SC+S16-(S1+S2+S3+S4+S5+S6+S7+S8+S9+S10+S11+S12+S13+S14+S15)=90.50mm2=0.905cm2
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.
2236	
2238	Calculated by the area of an approximate ellipse
2247	by Vernier Calliper
2250	Measurement by calliper. Area calculation: hollow cylinder and a summary of rectangular solids.
2255	Area calculated by considering Parabola & circle
2256	
2265	1X mathematical surface calculation with digital calliper 1X digital surface calculation with App
2284	Drawing the outline on gird paper,then calculate the area by the ratio of weight to area.
2289	Suppose the main body part of leaf is a ellipse and the tail part of the leaf is a circle. Calculate all surface area including thickness. Then subtract the surface area of hollow parts to obtain the final surface area of the sample.
2290	
2293	Using graph paper, trace the sample and upscaling it.
2295	3D scanner
2310	Calculated surface area of ellipse and circle by using Vernier calliper.
2311	Consider four triangle surface area =1/2 X base X Hight X 4 =1/2 X 0.52 X 0.63 X 4 =0.655 cm2
2326	Divide the whole product into two parts(one is triangle and other one is trapezium). Consider cavities as rectangle & upper parts of pendant consider outer and inner cavity circle also take thickness of pendant by considering it rectangle (l X w).
2330	Digital calliper
2347	/
2350	It was calculated by the sum of each part.
2352	Double sided leaf area minus the area of the hollowed out part plus the side area and the area of the hollowed out inner edge
2357	
2363	none
2365	
2366	
2369	
2370	
2373	Compare the blade to an ellipse, subtract the area of the hollow part (similar to circular, elliptical, triangular, trapezoidal), and add the surface area of the hollow part and the outer ring side of the blade.
2375	We measured on millimetre paper. We used digital calliper to measure the side surface areas.
2377	using common geometrical shapes
2378	
2379	By Vernier calliper.
2380	This article considered as two triangles that formula is (axh)/2. Here a=base, h=height & one rectangle that's formula is AxB, Here A=Length, B=Width. Top part considered circle that formula is □r² Here r=radius.
2381	In this pendant we found ellipse, circle and cylinder.Then measure the area and added.Finally minus with the gap area and found 0.832 cm2.
2385	
2406	Gravimetric method using graph paper with magnifying.
2429	Divide the sample into several regular shaped parts, calculate the surface area
2449	Calculated the are by calaulating the area of small areas and then adding
2459	Area was calculated by graph methodology considering whole leaf (sample). Average value of 04 measurements are taken.
2475	We use a graph paper to calculate the surface: we draw the pendant on the graph paper. We calculate the number of sqare. We obtain the surface for one face. After we multiplicate by 2 to obtain the total surface of the pendant.

lab	description how the surface was measured and calculated
2482	Sample surface was copied and magnified, copy was cut out and weighed against paper with known area. Girth was measured by using wire on all edges after fixing the magnified copy with needles and scaled down. Thickness was measured with a digital calliper. Area (surface) = 0,2671 cm²; Girth = 10 cm; Thickness = 0,24 mm (2 * 0,2671) cm² + 10 cm * 0,024 cm = 0,7742 cm
2511	
2522	Our lab used the digital vernier calliper.
2538	
2573	
2582	The pendent roughly devided into different shapes (Triangle, Rectangles, Trapezoid and calculate the surface area and got the total)
2590	
2602	Scan flat object with millimeterpaper as background. Digitaly Zoom in sufficiently and print enlarged image of object with millimeterscale on paper. Cut defined rectangular area around the object and weigh. Then cut the image of the object out and weigh. Surface area is proportional to paperweight.
2624	
2637	
2652	
2666	I divided the object into four triangles, added them together, subtracted the empty space, then multiplied by two for the 2 faces
2671	Using a Digital Vernier Calliper.
2674	measured by vernier calliper calculated by calculator
2678	
2703	The item was treated as a series of shapes such as an ellipsoid, cuboid, triangle or circle. Each shape had the surface area calculated and then added or subtracted accordingly.
2719	
2720	Calculate by converting leaves into regular shapes through filling methods
2737	Calculate the overall area first, then deduct the area of the hollow part
2818	
2829	
2864	Draw on a 10*10 mm2 drawing, calculate the grid.
2867	Parts of the samples were divided into several shape rules, calculating the surface area.
2900	
2912	The area of flat surface is measured with graph paper. The area of side surfaces is measured with calibrated calliper.
2977	
2989	Firstly take Circle on upper side. Then mark the oval of over all leave. Use edges outside the oval Half Circle. Take all cavity using formaula of Circle, Trapazium, Triangle & Rectangles. We subracted all Cavity from surface area. After the sum of all area to get 0.63 cm2
3100	Use a vernier calliper to calculate the area,treat the sample as an ellipse,and then subtract the area of the hole.
3110	
3116	The area of each part was calculated by assuming their respective common geometrical shapes and each dimension was measured by calliper.
3118	the sample is copied with 400% magnification,then the sample is placed on millimeter block paper. the result of duplicating the sample in millimeter blocks is then calculated for its area. calculate the area of the thick part of the sample. the final result is the area of the upper+bottom+thick ar
3134	Scanning, magnifying, cutting perimetrically (empty spaces cut out), weighting and comparing its mass with mass of sheet of known area.
3153	The dimensions are measured by digital calliper. The surface area of the pendant is calculated as two oval shapes. The empty space is subtracted as different shapes including circle, rectangle, triangle and trapezoid. The surface area of the thickness of the outer and inner edge is then added.
3172	
3182	Using Equation 1/2 x W x H for 9 point. Using Equation 1/2 x (parallel side effect) x H for 2 point. Using Equation 2 x 3.14 x r x h for 1 point. Using Equation W x L for 11 point. Using Equation 3.14 x (R2 - r2) for 1 point. Using Equation (3.14/4) x D x d for 2 point.
3185	Calculation after manual measurement with digital display vernier calliper
3190	First,calculate the area of the sample as an ellipse,and then subtract the area of the hollowed out rectangles and circles,And then multiply by 2 to get the final result.
3197	Surface area of sample was calculated using formula of a few rectangular prisms.
3210	
3218	Total area = Leaf area + Connecting ring - hollowing=0.998cm2 + 0.050cm2 - 0.102cm2=0.946cm2
3225	Use a grid paper with defined area as reference.
3228	Measure the overall length/width and the length/width of the hollowed-out part, then use the cut-and-patch method to turn it into a rectangle to calculate the area.
3230	Area of leaf pendant = [Area of Major Leaf Shape + Area of circle (at the base of the leaf)] – Area of 15 inner designs on leaf = [0.387550 + 0.0282884] – 0.1768) = 0.2390384 cm2
3237	

APPENDIX 5

Number of participants per country

3 labs in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
1 lab in CAMBODIA
4 labs in FRANCE
8 labs in GERMANY
1 lab in GREECE
1 lab in GUATEMALA
8 labs in HONG KONG
4 labs in INDIA
2 labs in INDONESIA
8 labs in ITALY
1 lab in KOREA, Republic of
1 lab in MAURITIUS
1 lab in MOROCCO
30 labs in P.R. of CHINA
4 labs in PAKISTAN
1 lab in POLAND
1 lab in SRI LANKA
1 lab in SWITZERLAND
2 labs in TAIWAN
2 labs in THAILAND
2 labs in TUNISIA
4 labs in TURKEY
2 labs in U.S.A.
2 labs in UNITED KINGDOM
5 labs in VIETNAM

APPENDIX 6

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

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)