

**Results of Proficiency Test
Nickel Release
May 2017**

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: dr. R.G. Visser
Correctors: ing. R.J. Starink & ing. A.S. Noordman-de Neef
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1 INTRODUCTION

Nickel has always been used in various applications, as a pure metal, as a plated substance on another metal or as an alloy. Nickel applications usually do not give problems, but when Nickel comes into prolonged and direct contact with the human skin, sensitization can occur. When a person becomes sensitive to Nickel, even the smallest amounts can provoke an allergic reaction. By this, Nickel is the most frequent cause of contact allergy in Europe. Both the contact itself (sometimes enhanced by damaged skin) and skin conditions as sweat can cause the body to be exposed to Nickel. In order to decrease the amount of people that become sensitized, Nickel containing items that are used in prolonged human contact are tested for Nickel release. These products involve products like jewellery in piercings (ear rings), other jewellery, watches or clothes fasteners, such as buttons and belts.

On request of several participants, the Institute of Interlaboratory Studies decided to organise an interlaboratory study for the determination of Nickel release in the annual testing program since 2014. This PT was continued each following year.

In the 2017 interlaboratory study 127 laboratories in 30 different countries did register for participation. See appendix 5 for the number of participants per country. In this report, the test results of the 2017 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 Spijkensisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send six pieces of one non-coated sample (labelled #17575), positive on Nickel release and a piece of chain (labelled #17576) for surface determination only. Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation. Also an inventory was made of the analytical details of the used test method, by means of a questionnaire, which was included in the report form.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkensisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation 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 March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

Nickel Release Determination

The samples were purchased from a local supplier and consisted of square metal pieces with a hole in one of the corners. The pieces were massive, prepared from one alloy and not plated or coated. The material was polished prior to the laser cutting. The dimensions of each sample were approximately 1.2 x 1.2 x 0.2 cm and the hole had a diameter of approx 2 mm. Samples were labelled #17575.

Twenty-four stratified randomly selected samples were tested using EN1811:2011 and single test results were averaged per three to check the homogeneity of the batch. The test results of the homogeneity tests, after exclusion of one clear outlying test result are shown in table 1.

	<i>Nickel release ($\mu\text{g}/\text{cm}^2/\text{week}$) averaged per 3</i>
sample #17575-1	0.22
sample #17575-2	0.32
sample #17575-3	0.23
sample #17575-4	0.31
sample #17575-5	0.24
sample #17575-6	0.23
sample #17575-7	0.24

Table 1: homogeneity test results of subsamples #17575

From the above test results the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2, in the next table:

	<i>Nickel release ($\mu\text{g}/\text{cm}^2/\text{week}$)</i>
r (observed)	0.11
reference	Horwitz
0.3 x R (reference)	0.05

Table 2: evaluation of the repeatability of subsamples #17575

The calculated repeatability was not in agreement with 0.3 times the corresponding reproducibility of the reference test method. Upon investigation it became clear that the polished surface of the test items was disrupted around the small hole, a phenomena that was not observed in previous sample preparations. This year the hole has been made smaller than in previous years (2mm vs 4mm). This may have caused the heat from the laser cutting to be concentrated in a relatively small area around the hole and this may have caused some

melting of the metal. As the observed variation RSD_r in the homogeneity results (15%) was smaller than the variations as observed in previous PTs, the suitability of the sub samples was assumed, provided that not 3 test items, but 6 test items were sent to the participating laboratories. When the participants would test all 6 items, the average of the 6 test results, after exclusion of an outlying and/or a suspect test result, would be sufficiently reliable.

Surface Determination

A chain was purchased from a local supplier. It was cut into pieces of chain, 6 links long, all with the same length and model. The samples were labelled #17576. No homogeneity tests were done because only surface determination has been requested for this sample.

Six items of sample #17575 and one item of sample #17576 were sent to each of the participating laboratories on May 10, 2017.

2.5 ANALYSES

The participants were requested to determine Nickel release on sample #17575 and the total surface only on sample #17576, applying the analysis procedure that is routinely used in the laboratory. However, in the letter of instructions the participants were requested to use at least three items for the determination of Nickel release. The other three items could be used for additional determinations if needed.

It was requested 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 calculations.

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 that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

3.2 GRAPHICS

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

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. EN reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of 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 targets values were used. In some cases a reproducibility based on former iis proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

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

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare.

The usual interpretation of z-scores is as follows:

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

4 EVALUATION

During the execution of this proficiency test no considerable problems were encountered. From the 127 participants, four participants reported test results after the deadline for reporting and five other participants did not report any test results at all. In total 122 laboratories reported 122 Nickel release test results. Observed were 14 outlying test results, which is 11%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER SAMPLE

In this section, the reported test results are discussed per sample. All statistical results reported on the sample are summarised in appendix 1. The abbreviations used in these tables are listed in appendix 6.

Test method EN1811:2011 does not have a true precision statement that mentions a repeatability and/or a reproducibility. In Annex A is mentioned that the measurement uncertainty in a 2008 interlaboratory study was 46%, while in Annex B is stated "The relative test method reproducibility in this ILC was 33.3%". Both variations could not be met by far in previous iis PTs. Therefore it was decided to use a target reproducibility derived from the Horwitz equation. This target is dependent on the measured nickel concentration and ranges from 62% at 0.3 $\mu\text{g Ni/cm}^2/\text{week}$ up to 37% at 10 $\mu\text{g Ni/cm}^2/\text{week}$.

Sample #17575: Nickel release:

The determination of Nickel release at a low concentration level of 0.26 $\mu\text{g}/\text{cm}^2/\text{week}$ was problematic. Fourteen statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the target reproducibility estimated from the Horwitz equation. The very low Nickel release level may (partly) explain the relatively large variation.

Sample #17576: Surface Determination:

The surface determination of the chain may be problematic. Four statistical outliers were observed in the reported range of 0.1974 – 14.97 cm^2 . No official test method exists for surface determination; therefore no hard conclusions could be drawn. However, the variation for this sample (6.7%) is very large in comparison with the variation in previous PT in which the surface determination was evaluated (1.7% - 4.9%) and with the variation of the surface determination on sample #17575 (1.3%).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as found for the group of participating laboratories and the target reproducibility estimated from the Horwitz equation in the next table:

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R (target)</i>
Nickel release	$\mu\text{g}/\text{cm}^2/\text{week}$	108	0.26	0.19	0.17
Contact surface	cm^2	111	3.89	0.14	n.a.

Table 3: reproducibilities of test results on sample #17575

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

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R (target)</i>
Surface Determination	cm^2	116	12.84	2.4	n.a.

Table 4: reproducibility of test results on sample #17576

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2017 WITH PREVIOUS PTS

	<i>May 2017</i>	<i>May 2016</i>	<i>May 2015</i>	<i>May 2014</i>
Number of reporting labs	122	125	123	111
Number of test results reported	122	124	119	222
Statistical outliers	14	8	11	4
Percentage outliers	11%	6.5%	9.8%	1.8%

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

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

In table 6 the observed uncertainties in this PT are compared with the uncertainties as observed in the previous PTs.

	<i>May 2017</i>	<i>May 2016</i>	<i>May 2015</i>	<i>May 2014</i>
Nickel Release	26%	18%	28%	27-31%
<i>Surface Determination</i>	1.3 - 6.7%	2.3 - 4.9%	1.7%	9 - 10%

Table 6: comparison of uncertainties (relative in %) of this PT and previous PTs

No quality improvement is visible in the Nickel Release determination as the uncertainty did not decrease compared to previous years, but the fact that the Nickel release level of the 2017 PT was much lower than in previous year, may (partly) explain this.

The uncertainty of the surface determination of sample #17576 (chain) is larger than of sample #16576 (tea spoon) and than of sample #17575 (square plate) which was to be expected for the more difficult chain sample that was used in the 2017 PT.

5 DISCUSSION OF REPORTED TEST METHOD DETAILS

Details of various analytical steps were requested to be reported, like the average volume of sweat simulant that was added to one piece of metal, the average surface of one piece of metal used for the calculation, the number of pieces of metal used for the Nickel release determination, which ratio in mL/cm² was used for the start solution versus the sample surface, whether the test vessel was pre-treated and how the surface of the chain was determined. These reported details are summarized in appendices 2 - 4.

Determination of contact surface of the square test items #17575:

In total 122 laboratories reported the average surface area used, see appendix 2. The reported average surface area for sample #17575 varied from 2.4 to 11.3 cm². After exclusion of eleven (10%!) statistically outlying data, the surface range narrowed from 3.76 to 4.067 cm². The observed RSD of 1.3% after the rejection of the eleven outliers is better than observed in all previous PT.

In this PT the overall RSD_{nickel release} for sample #17575 is 26%. This is the sum of the variation in contact surface determination and the variation in the Nickel determination. It can be concluded that the variation in the surface determination of this (simple squared) object does not affect the overall variation of the Nickel release determination.

Volume of the start solution:

It was observed that a number of participants were confused about the question: "Average volume added to one piece of metal", see appendix 2. Several participants reported the end volume after dilution, e.g. 10 mL. The test method of EN1811:2011 prescribes that the amount of the start test solution to be used should be 1 ml per cm² surface area, which is in this PT about 4 ml per test item. Not all participants used this ratio. One participant gave as reason "Standard ratio 1 ml/cm² was not enough to immerse sample; usage of disposable vessels (no cleaning)".

However, the majority of the participants (87%) reported a ratio of approx. 1 ml/cm². The range of used ratios was 0.52 – 7.9 ml/cm². The range of initial volumes was 2.4 – 11.3 ml.

Number of test items #17575 used for the Nickel release determination:

It is remarkable to see that in spite of the explicit instruction to use at least 3 test items (as per EN1811), no less than 7 participants used only 1 (one) test item and one participant only two test items, see appendix 2. Several participants observed a relatively large variation in the test results, but only 25 participants used all 6 test items; only 6 used 5 of them and 75 participants did use 3 test items as per EN1811. This may well explain the relatively large variation observed in this PT. Two participants reported also the 6 individual test results on each of the test items. In each series of 6 test results deviating test results are present, but both laboratories decided not to exclude deviating test results, but to report the average of all six test results. This decision resulted twice in an average test result with high z-score. It is expected that the variation in this PT would have been smaller when all participants had tested all 6 test items and had rejected one or two deviating test results. It is advised to do so in every day's practice when testing consumer articles. Consumer articles from the market, especially plated ones, that are to be tested on Nickel release show much more variation than the PT test items as used in the iis PTs. And therefore a reliable conclusion cannot be drawn on a single Nickel release determination on only one test item and often not even on the average test result of a triplicate determination as per EN1811. This will be important especially when the Nickel release is close to the rejection limit.

Pre-treatment of vessel:

The vessel, used for leaving the sample in the sweat solution for a week, should be pre-treated with 5% Nitric acid for at least 4 hrs, see paragraph 6.4 of EN1811:2011. This is done to remove any Nickel present from earlier use. About 57% the participants (70) reported to have done a pre-treatment, of which 55 with 5% (or higher) HNO₃ for at least 4 hours, but 31% of the participants (38) did not use any pre-treatment (11% did not answer to this question), see appendix 3.

When no pre-treatment is used, there will be a risk that the test result for Nickel release will be higher than correct. To check whether some effect is visible, the test results of the laboratories that did not use any pre-treatment were compared with the test results after treatment with diluted nitric acid of at least 4 hrs., see table 7.

	<i>No pre-treatment</i>	<i>≥ 5%HNO₃ pre-treatment for ≥4hrs</i>
Number of test results	35	49
Statistical outliers	3	6
Average	0.27 µg/cm ² /week	0.26 µg/cm ² /week
Standard deviation	0.076 µg/cm ² /week	0.056 µg/cm ² /week
RSD%	28%	22%

Table 7: influence of pre-treatment of test vessel

The effect of the acid pre-treatment of the vessel is visible, mainly in the variation. The variation in the test results from a vessel that was not pre-treated is higher than the variation in the test results from a correctly pre-treated test vessel. Quality improvement may be possible for this parameter.

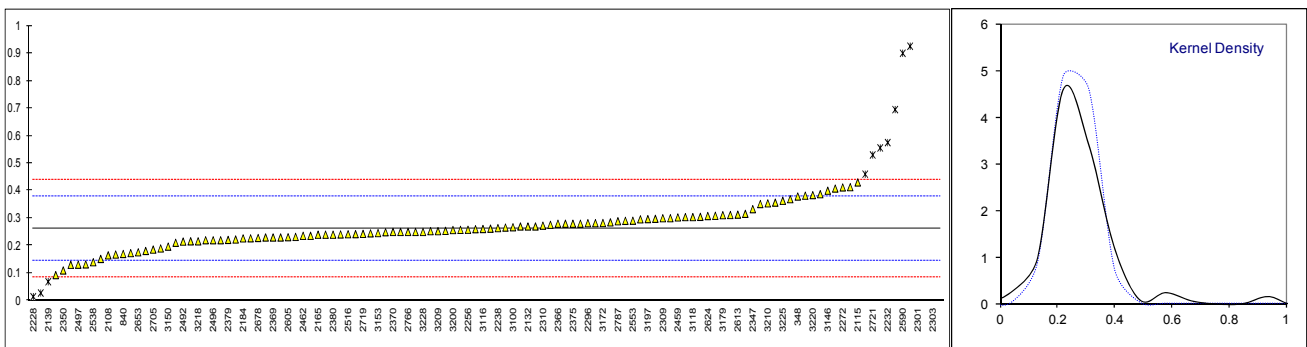
APPENDIX 1Determination of Nickel Release on sample #17575; result in $\mu\text{g}/\text{cm}^2/\text{week}$

lab	method	value	mark	z(targ)	remarks
213		-----		-----	
339	EN1811 + AC	0.9255	R(0.01)	11.21	
348	EN1811 + AC	0.379		1.97	
362	EN1811	0.028	R(0.01)	-3.96	
551		0.19		-1.22	
622	EN1811	0.3692		1.81	
623	EN1811:11 + A1	0.3516		1.51	
840	EN1811	0.17		-1.56	
841	EN1811	0.23		-0.55	
2108	EN1811	0.165		-1.64	
2115	EN1811	0.43		2.83	large variation: 1.0, 0.27, 0.41, 0.45, 0.35 and 0.43 $\mu\text{g}/\text{cm}^2/\text{week}$
2117	EN1811	0.181		-1.37	
2121		-----		-----	
2129	EN1811	0.413		2.55	
2132	EN1811 + AC	0.27		0.13	
2137	EN1811:15	0.132		-2.20	
2139	EN1811	0.069	R(0.01)	-3.27	
2165	EN1811	0.240		-0.38	
2184	EN1811 + AC	0.2268		-0.60	
2190	EN1811	0.22	C	-0.72	first reported 0.14
2201	EN1811	0.253		-0.16	
2213	EN1811	0.28		0.30	
2228	EN1811	0.0144	C,R(0.01)	-4.19	first reported 0.0286
2232	EN1811	0.575	R(0.05)	5.28	
2238	EN1811 + AC	0.264		0.03	
2247	EN1811	0.25		-0.21	
2255	EN1811	0.304		0.70	
2256	EN1811 + AC	0.258		-0.07	
2265	In house	0.695	R(0.05)	7.31	
2272	EN1811	0.412		2.53	
2289	EN1811 + AC	0.254		-0.14	
2290	EN1811	0.285		0.38	
2293	EN1811	0.3117		0.83	
2295	EN1811	0.24	C	-0.38	first reported 0.2
2296	EN1811	0.282		0.33	
2301	EN1811	1.1188	C,R(0.01)	14.47	first reported 0.559
2303	EN1811	1.158	R(0.05)	15.13	
2309	EN1811 + AC	0.30		0.64	
2310	EN1811 + AC	0.273		0.18	
2311	EN1811 + AC	0.276		0.23	
2330	EN1811	0.2269		-0.60	
2347	EN1811 + AC	0.333		1.19	
2350	EN1811 + AC	0.110		-2.57	
2352	EN1811 + AC	0.2226		-0.67	
2357	EN1811 + AC	0.242		-0.34	
2362	EN1811	0.266		0.06	
2363	EN1811 + AC	0.241		-0.36	
2365	EN1811	0.249		-0.23	
2366	EN1811 + AC	0.280		0.30	
2369	EN1811	0.23		-0.55	
2370	EN1811	0.25		-0.21	
2375	EN1811 + AC	0.28		0.30	
2379	EN1811	0.221		-0.70	
2380	EN1811 + AC	0.2402		-0.37	
2385	EN1811 + AC	1.13	R(0.01)	14.66	
2390	EN1811	0.173		-1.51	
2403	EN1811 + AC	0.270		0.13	
2410	EN1811	0.27		0.13	
2429		-----		-----	
2432	EN1811	0.2106		-0.87	
2442	EN1811 + AC	0.216		-0.78	
2459	EN1811 + AC	0.303		0.69	
2462	EN1811 + AC	0.236		-0.44	
2475	EN1811 + AC	0.316		0.91	
2489	EN1811	0.23		-0.55	
2492	EN1811 + AC	0.215		-0.80	
2495	EN1811	0.4079		2.46	
2496	EN1811	0.22		-0.72	
2497	EN1811 + AC	0.131		-2.22	
2500	EN1811 + AC	0.26		-0.04	
2511	EN1811	0.245		-0.29	
2514	EN1811	0.3088		0.79	
2516	EN1811	0.241666		-0.35	
2538	EN1811	0.14		-2.07	2 from 6 results were rejected

2553	EN1811	0.291		0.48	
2560	EN1811	0.2575		-0.08	
2563	EN1811	0.46	R(0.05)	3.34	
2567	EN1811	0.298		0.60	
2590	EN1811:11 + A1	0.9	C,R(0.01)	10.77	first reported 0.2610
2605	EN1811 + AC	0.231		-0.53	
2613	EN1811	0.313		0.86	
2624	EN1811	0.308		0.77	
2629	EN1811	0.130548		-2.23	
2653	EN1811	0.176	C	-1.46	first reported 11.636 acc. to EN16128:11
2657	EN1811	0.28		0.30	
2666	EN1811	0.5556	R(0.05)	4.96	
2674	EN1811	0.2905		0.48	
2678	EN1811	0.228		-0.58	
2705	EN1811	0.186		-1.29	
2713	EN1811	0.093		-2.86	
2719	EN1811	0.243		-0.33	
2721	EN1811/EN1822	0.53	R(0.05)	4.52	large variation: 0.33, 0.28, 0.94, 0.12, 0.22 and 1.3 µg/cm2/week
2737	EN1811 + AC	0.3874	C	2.11	first reported 1.1888
2741	EN1811	0.262		-0.01	
2743	EN1811 + AC	22.74963	R(0.01)	379.96	
2758	EN1811	0.382		2.02	
2766	EN1811	0.25		-0.21	
2783	EN1811	0.3567		1.59	
2787	EN1811	0.289		0.45	
3100	EN1811	0.2672		0.08	
3110	EN1811	0.30		0.64	
3116	EN1811 + AC	0.2608		-0.03	
3118	EN1811	0.304		0.70	
3146	EN1811	0.400		2.33	
3150	EN1811	0.1968		-1.11	
3153	EN1811 + AC	0.2459		-0.28	
3154	EN1811	0.152		-1.86	
3160	EN1811 + AC	0.296		0.57	
3172	EN1811 + AC	0.2825		0.34	
3176	EN1811	0.250	C	-0.21	first reported 0.508
3179	EN1811	0.311		0.82	
3182	EN1811	0.22		-0.72	
3183	§64 LFGB B82.02-6	0.282		0.33	RSD = 31%
3185	EN1811 + AC	0.232		-0.51	
3191	EN1811	0.305		0.72	
3197	EN1811 + AC	0.29670		0.58	
3200	EN1811	0.257		-0.09	
3209	EN1811	0.253		-0.16	
3210	EN1811	0.354		1.55	
3214	EN1811	0.236		-0.44	
3218	EN1811	0.216		-0.78	
3220	EN1811	0.384	C	2.06	first reported 10.9
3225	EN1811	0.36355		1.71	
3228	EN1811	0.25		-0.21	
3233		----		----	
3237	EN1811	0.168		-1.59	
8008		----		----	

normality OK
 n 108
 outliers 14
 mean (n) 0.262
 st.dev. (n) 0.0673 =26%
 R(cal.) 0.189
 R(Horwitz) 0.166

Compare R(EN1811:2011) = 0.087

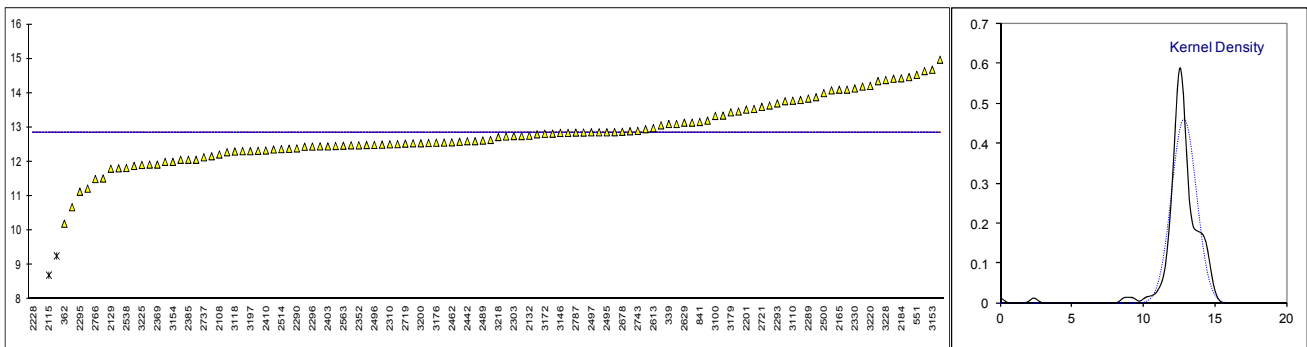


Determination of Surface determination on sample #17576; results in cm²

lab	method	value	mark	z(targ)	remarks
213		----		----	
339	see appendix 4	13.1	C	----	first reported 2.37
348	see appendix 4	14.3477		----	
362	see appendix 4	10.2		----	
551	see appendix 4	14.53		----	
622	see appendix 4	2.33	C,R(0.01)	----	first reported 2.38
623	see appendix 4	13.464		----	
840		----		----	
841	see appendix 4	13.16		----	
2108	see appendix 4	12.2145		----	
2115	see appendix 4	8.7	R(0.01)	----	reported 1.45 x 6 = 83.7(?)
2117	see appendix 4	12.5		----	
2121		----		----	
2129	see appendix 4	11.80		----	
2132	see appendix 4	12.76		----	
2137	see appendix 4	12.56		----	
2139	see appendix 4	14.97		----	
2165	see appendix 4	14.10		----	
2184	see appendix 4	14.43		----	
2190		----		----	
2201	see appendix 4	13.52		----	
2213	see appendix 4	13.88		----	
2228	see appendix 4	0.019752	R(0.01)	----	
2232	see appendix 4	14.466		----	
2238	see appendix 4	12.48		----	
2247	see appendix 4	12.6		----	
2255	see appendix 4	12.38		----	
2256	see appendix 4	13.764		----	
2265	see appendix 4	12.72		----	one peace(part) of chain is 2,12 cm ²
2272	see appendix 4	12.2782		----	
2289	see appendix 4	13.84		----	
2290	see appendix 4	12.39		----	
2293	see appendix 4	13.7		----	
2295	see appendix 4	11.13		----	
2296	see appendix 4	12.444		----	
2301	see appendix 4	12.820		----	
2303	see appendix 4	12.75		----	
2309	see appendix 4	12.54		----	
2310	see appendix 4	12.51		----	
2311	see appendix 4	12.547		----	
2330	see appendix 4	14.1357		----	
2347	see appendix 4	12.45		----	
2350	see appendix 4	12.637		----	
2352	see appendix 4	12.48		----	
2357	see appendix 4	11.92		----	
2362	see appendix 4	14.08		----	
2363	see appendix 4	11.88		----	
2365	see appendix 4	12.06		----	
2366	see appendix 4	12.862		----	
2369	see appendix 4	11.92		----	
2370	see appendix 4	12.95		----	
2375	see appendix 4	12.06		----	
2379		----		----	
2380	see appendix 4	13.2		----	
2385	see appendix 4	12.06		----	
2390	see appendix 4	13.1		----	
2403	see appendix 4	12.45		----	
2410	see appendix 4	12.33		----	
2429		----		----	
2432	see appendix 4	12.31		----	
2442	see appendix 4	12.6		----	
2459	see appendix 4	12.95		----	
2462	see appendix 4	12.568		----	
2475	see appendix 4	12.89		----	
2489	see appendix 4	12.62		----	
2492	see appendix 4	12.583		----	
2495	see appendix 4	12.86		----	
2496	see appendix 4	12.49		----	
2497	see appendix 4	12.8585		----	
2500	see appendix 4	14.0		----	
2511	see appendix 4	13.8		----	
2514	see appendix 4	12.37		----	
2516	see appendix 4	11.51595		----	
2538	see appendix 4	11.82		----	
2553	see appendix 4	9.26	C,R(0.01)	----	first reported 4.6342
2560	see appendix 4	12.8526		----	

2563	see appendix 4	12.47	----	----
2567	see appendix 4	12.36	----	----
2590	see appendix 4	11.810	----	----
2605	see appendix 4	14.64	----	----
2613	see appendix 4	12.98	----	----
2624	see appendix 4	12.80	C	---- first reported 2.14
2629	see appendix 4	13.14	----	----
2653	see appendix 4	12	----	----
2657	see appendix 4	14.1	----	----
2666	see appendix 4	10.680	----	----
2674	see appendix 4	14.42	----	----
2678	see appendix 4	12.875	----	----
2705	see appendix 4	13.639	----	----
2713	see appendix 4	12.162	C	---- first reported 2.027
2719	see appendix 4	12.53	----	----
2721	see appendix 4	13.6	----	----
2737	see appendix 4	12.133	C	---- first reported 12.4515
2741	see appendix 4	12.32	----	----
2743	see appendix 4	12.90110	----	----
2758	see appendix 4	13.345	----	----
2766	see appendix 4	11.5	C	---- first reported 7.536
2783	see appendix 4	12.8592	----	----
2787	see appendix 4	12.8508	----	----
3100	see appendix 4	13.3353	----	----
3110	see appendix 4	13.779	----	----
3116	see appendix 4	14.19	----	----
3118	see appendix 4	12.2996	----	----
3146	see appendix 4	12.84	----	----
3150	see appendix 4	12.51345	----	----
3153	see appendix 4	14.68	----	----
3154	see appendix 4	12	----	----
3160	see appendix 4	13.062	C	---- first reported 1.9964
3172	see appendix 4	12.817	----	----
3176	see appendix 4	12.556	----	----
3179	see appendix 4	13.44	----	----
3182	see appendix 4	12.44	C	---- first reported 10.15
3183	see appendix 4	12.488	----	----
3185	see appendix 4	13.14	----	----
3191	see appendix 4	12.84	----	----
3197	see appendix 4	12.31	----	----
3200	see appendix 4	12.54	----	----
3209	see appendix 4	12.46	----	----
3210	see appendix 4	11.22	----	----
3214	see appendix 4	13.538	----	----
3218	see appendix 4	12.721	----	----
3220	see appendix 4	14.209	----	----
3225	see appendix 4	11.91	----	----
3228	see appendix 4	14.38	----	----
3233	see appendix 4	12.75	----	----
3237	see appendix 4	12.738	----	----
8008		----	----	----

normality OK
n 116
outliers 4
mean (n) 12.84
st.dev. (n) 0.857 = 6.7%
R(calc.) 2.40
R(target) n.a.



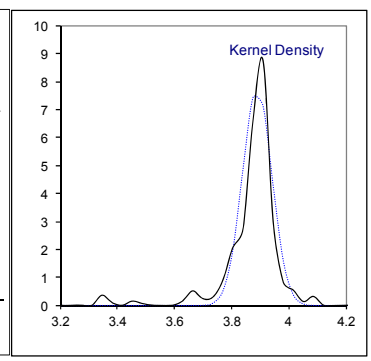
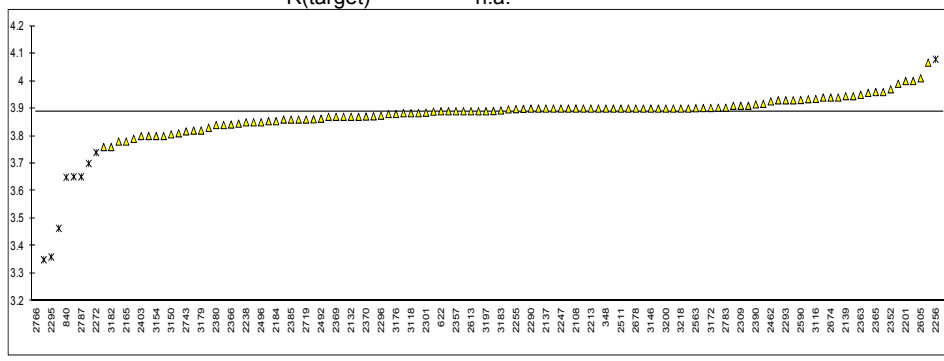
APPENDIX 2

Average volumes added, average surfaces and number of pieces used of sample #17575

lab	average volume of sweat simulant added to one item (ml)	average surface of one item used (cm ²)	number of items used for Ni-release determination			
213	----	----	----			
339	3.8	3.76	3			
348	5	3.9	3			
362	11.3	11.3	3			
551	----	3.91	3			
622	4	3.89	3			
623	5	3.89	3			
840	4	3.65	3			
841	3.9	3.9	6			
2108	8.3333333333	3.9	3			
2115	3.85	3.85	----			
2117	5.5	3.88	3			
2121	----	----	----			
2129	4	3.87	3			
2132	4	3.87	5			
2137	4	3.90	3			
2139	4	3.945	6			
2165	4	3.78	3			
2184	4	3.854	3			
2190	5	C, fr. 0.3	3.9	C, fr.0.21	1	C, fr.0.13
2201	4.0	4.0	6			
2213	4	3.9	3			
2228	5	C,fr.10	3.8539	3		
2232	4.0	3.917	3			
2238	3.9	3.85	6			
2247	4	3.9	6			
2255	5.0	3.898	3			
2256	5	4.079	R(0.05)	3		
2265	5	3.87		2		
2272	10	3.74	R(0.05)	1		
2289	4.0	4.0		6		
2290	3.9	3.9		6		
2293	25	3.93		3		
2295	5	C, fr.10	3.36	R(0.01)	3	
2296	2	3.874		3		
2301	4	C, fr.7.50	3.885		6	
2303	4	3.99		3		
2309	4	3.91		3		
2310	4	C,fr.10	3.81		3	
2311	5	3.957		3		
2330	5	C, fr.2.5	3.8718		3	
2347	3.93	3.93		3		
2350	4	C, fr.10	3.889		3	
2352	4.00	3.97		1		
2357	4	3.89		6		
2362	4	3.9		4		
2363	4	3.95		3		
2365	4.0	3.96		6		
2366	3.842	3.842		6		
2369	3.9	3.87		3		
2370	4	3.871		3		
2375	3.87	C, fr.10	3.87		1	
2379	5	3.90		1		
2380	4.0	3.84		3		
2385	6	3.86		5		
2390	3.9	3.915		3		
2403	3.80	3.80		3		
2410	4	3.86		6		
2429	----	----	----	----		
2432	4	C, fr.10	3.78		5	
2442	3.7	C, fr.10	3.7	R(0.05)	3	
2459	4	3.9		6		
2462	4.000	3.926		3		
2475	3.93	3.93		3		
2489	3.89	C, fr.10	3.89		3	
2492	3.0	3.863		3		
2495	4.00	3.82		3		
2496	3.85	3.85		3		
2497	10	3.4642	R(0.01)	3		
2500	5	3.9		3		
2511	3.9	3.9		3		
2514	5.0	3.899		3		

lab	average volume of sweat simulant added to one item (ml)	average surface of one item used (cm ²)	number of items used for Ni-release determination
2516	3.6516	3.6516	R(0.05) 6
2538	4	3.91	6
2553	6.00	3.8451	3
2560	4	3.883	3
2563	4	3.901	6
2567	4	3.86	4
2590	10.0	is final volume	3.931 6
2605	4.01	4.01	6
2613	3.90	3.89	3
2624	4	C, fr.10	3.94 3
2629	5		3.83 5
2653	6.2	fr.6	3.350 R(0.01) 3
2657	4		3.8 3
2666	4	C, fr.16	3.90 3
2674	4		3.94 6
2678	3.9		3.9 3
2705	4	C, fr.10	3.840 6
2713	5	C, fr.10	3.891 3
2719	4		3.86 3
2721	5.2		3.9 6
2737	4.0		3.9018 C, fr.39534 3
2741	4	C, fr.9	3.96 3
2743	4		3.8169 4
2758	7.00		3.903 3
2766	10		2.4 R(0.01) 1
2783	4.0		3.903 3
2787	4		3.6516 R(0.05) 6
3100	3.9339		3.9339 3
3110	4		3.8831 3
3116	4		3.935 6
3118	5		3.883 3
3146	3.9		3.9 6
3150	4		3.8062 3
3153	3.9		3.861 6
3154	30		3.8 -----
3160	5	C, fr.10	3.945 C, fr.3.972 3
3172	3.9		3.902 4
3176	5		3.88 3
3179	5 and 10		3.82 4
3182	5	C, fr.10	3.76 3
3183	5		3.893 5
3185	4.0		3.89 3
3191	3.9		3.9 1
3197	3.9		3.89 3
3200	3.90		3.90 3
3209	3.9		3.94 3
3210	10		4.067 3
3214	3.9		3.90 5
3218	3.90		3.90 3
3220	7		3.79 3
3225	4		3.8 3
3228	3.90		3.90 3
3233	----		-----
3237	8		3.897 4
8008	----		-----
	normality	suspect	
	n	111	
	outliers	11	
	mean (n)	3.888	
	st.dev. (n)	0.05157	=1.3%
	R(calc.)	0.144	
	R(target)	n.a.	

RSD=31%



APPENDIX 3

Reported analytical details for sample #17575

lab	Was the test vessel pre-treated?	How many hours was the test vessel cleaned?	Solution that was used for cleaning	Ratio in mL/cm ² used for the start solution versus the sample surface?
213				
339	yes	1 hr	Nitric acid 20%	1/1
348	no			1.25 approximately
362				
551				
622	no			
623	yes	Overnight	HNO3	1:1
840	yes	4 hrs	Diluted nitric acid	1/1
841				
2108	no			
2115	no	1 week		1:1
2117	no			1,42
2121				
2129	no			1
2132	no			
2137	no			1 mL/cm ²
2139	yes	4 hrs		1.01 versus 1.0
2165	no			1.06:1
2184	no			1:1
2190	no			
2201	yes	4 hrs	5% nitric acid	1ml per 1cm ²
2213	yes			
2228	yes	24 hrs	artificial sweat solution	10 mL/cm ²
2232	yes	4 hrs	diluted HNO3 (5% <i>m/m</i>)	1:1
2238	yes	24 hrs	5% <i>HNO3</i>	1
2247				
2255	yes	4 hrs	5 % Nitric Acid	1 : 1.28
2256	yes	8 hrs	5% nitric acid	1.23
2265	no	168 hrs		1/1
2272	yes	overnight		5
2289	yes	8 hrs	10% <i>HNO3</i>	1:1
2290				
2293	yes	4 hrs	nitric acid	25 mL /3.0 - 5.0 cm ²
2295	yes	1 minute		
2296	no			
2301	yes	4 hrs	Nitric Acid	10:1
2303	yes	12 hrs	Laboratory detergent	1:1
2309	no			1:1
2310	yes	4 hrs	Dilute nitric acid	1 ml/cm ²
2311	yes	4hrs	5% Nitric acid	1:1
2330	yes	24 hrs	20% Nitric acid	1:1
2347	yes			
2350				
2352	yes	4hrs	20% Nitric acid	1:1
2357	yes	12 hrs	5% <i>HNO3</i>	1:1
2362	yes	4 hrs	Dilute Nitric acid	4 ml / 3.90 cm ²
2363	yes	4 hrs	5% <i>HNO3</i>	1:1
2365	yes	4 hrs	5% <i>HNO3</i>	1:1
2366	yes	12 hrs	1:1(V/V) <i>HCl</i> solution	1ml: 1cm ²
2369	yes	> 4 hrs	1:1 <i>HNO3</i> SOLUTION	
2370	yes	1 hr	10% <i>HNO3</i>	1.01 mL/ cm ²
2375	no			1-1
2379	yes	2 hr	5% <i>HNO3</i>	4 ml / 3.90 cm ²
2380	yes	5 hrs	5 % <i>HNO3</i>	1:1
2385	no	We use PP centrifuge tubes with screw caps (single use).		
2390	no			1 : 1
2403	yes	5 hrs	5% nitric acid	1:1
2410	no			1:1
2429				
2432				
2442	no			10 ml solution for 3.7 cm ²
2459				
2462	yes	12 hrs	5% Nitric Acid	1
2475		5 hrs	<i>HNO3</i> 5%	1
2489	no			1:1 ratio
2492	no			1ml to 1cm ²
2495	no			1.05
2496	yes	24 hrs	20% <i>HN03</i>	1:1
2497	yes	8 hrs	nitric acid	3:1
2500	yes	1 hr	DI Water	1.28
2511				

lab	Was the test vessel pre-treated?	How many hours was the test vessel cleaned?	Solution that was used for cleaning	Ratio in mL/cm ² used for the start solution versus the sample surface?
2514	yes	4 hrs	5% HNO3	1.28:1
2516	yes	4 hrs	5% nitric acid	1:1
2538	yes	12 hrs	5 % HNO3	4 ml / 3.91 cm ² = about 1:1
2553	yes	2 hrs		2:1
2560	yes	8 hrs	Nitric Acid	1:1
2563	yes	4 hrs	5% HNO3	1,03
2567	yes	12 hrs	5% HNO3	1:1
2590	no			2.56
2605	yes	4 hrs	5% HNO3	1:1
2613	yes	4 hrs	5% Nitric Acid	1:1
2624	no	4 hrs	HNO3 5%	1:1
2629	yes	24 hrs	HNO3 30%	
2653	no			
2657	no			1 : 1
2666	yes	24 hrs	nitric acid 3%	4
2674	yes	4 hours	5% nitric acid	1:1
2678	no			1:1
2705	no	0.5 hrs	5% HNO3	3
2713	yes	4 hrs	%5 (w/w) Nitric acid	1 mL for each cm ²
2719	no			1
2721	yes	4 hrs	5 % HNO3	ca. 1.3
2737	yes			
2741	yes	12 hrs	Nitric acid	2.2
2743	yes	0.25 hrs	Nitric acid	about 1
2758	no			2
2766	no			1 ml/cm2
2783	yes	3-4 rinses until clean	lab grade water	
2787	yes	5 hrs	5% HNO3	1.08
3100	yes	4 hrs	5% Nitric acid	1:1
3110				
3116	yes	> 4 hrs	Diluted nitric acid	1:1
3118				5 mL/ 3.88 cm2
3146	yes	4 hrs	nitric acid 5%	1:1
3150	no			~ 1:1
3153	yes	4 hrs	5% Nitric Acid	1 ml per cm2
3154				
3160	no			1.25
3172	no			1
3176	yes	1 day	HNO3	1
3179	no			1:1,3 resp. 1:2,6
3182	yes	24 hrs	10 % Nitric acid	1:1
3183	no			0,64
3185	yes	4 hrs	Dilute nitric acid	1:1
3191	yes	3 hrs	5% nitric acid	1:1
3197				1:1
3200	yes	24 hrs	5%HNO3	1:1
3209	yes	4 hrs	D.I. Water	1:1
3210	no			2.5
3214	yes	16 hrs	10% HNO3	1
3218	yes	4 hrs	5% HNO3	1:1
3220	yes	4 hrs	5% Nitric acid	2:1
3225	yes	4 hrs	5% HNO3	1:1
3228	no			1:1
3233				
3237	yes		%5 HNO3	2,053
8008				

APPENDIX 4

Reported Surface Determination details for sample #17576

lab	How was the surface of the chain measured and calculated?
213	
339	Use of electronic calipers square for determining the diameter and length of one plastic piece
348	For each chain link, surface is calculated using cylindrical approach. Length is measured with caliper and help of a thread, diameter with caliper directly. Total chain surface is calculated as sum of all chain links surfaces.
362	
551	
622	Determination of area, sample length is measured by using yarn and Diameter measured with caliper
623	
840	
841	
2108	$(2 \cdot \pi \cdot r \cdot h) \cdot 6 \text{ parts} = (2 \cdot 3,1415926 \dots \cdot 0,09 \cdot 3,6) \cdot 6 = 12,2 \text{ cm}^2$
2115	
2117	Surface determination of one chain link (by bending to a cylindric shape), multiplication with six
2121	
2129	-
2132	Vernier caliper was used
2137	I measured diameter by vernier calipers. I use a fiber to measure length.
2139	Using thread and digital caliper , calculate the area as follows: one piece of chain: $\pi \cdot r^2 \cdot 2$ (both sides) : $r^2 \cdot 3.14 \cdot 2 = 0.0905 \cdot 0.0905 \cdot 3.14 \cdot 2 = 0.051$ $\pi \cdot r \cdot h$ column : $2 \cdot \pi \cdot r \cdot h$ x height = $2 \cdot 0.0905 \cdot 3.14 \cdot 4.3 = 2.444$ One piece of chain = $\pi \cdot r^2 \cdot 2 + \pi \cdot r \cdot h = 0.051 + 2.444 = 2.495 \text{ cm}^2$ Total sample surface = $2.495 \cdot 6 = 14.97 \text{ cm}^2$
2165	using a cotton thread to measure the length of one single chain, then treat it as a cylinder.
2184	measure the diameter with digital caliper; measure the length with the help of thread
2190	
2201	Treat each piece as cylinder
2213	By vernier calliper
2228	The item was considered has a single ellipse with thickness. Then the total area was multiplied by 6.
2232	the chain calculated as a cylinder shape
2238	One clasp in the chain is modelled as a cylinder and its area is calculated.
2247	Used Vernier calliper for Dia and thread for length
2255	
2256	Measure the length of circle by cotton thread and caliper, and the diameter with caliper. Length (L) = 4.087cm Diameter (D) = 0.175 cm, surface area of circle = $\pi \cdot D \cdot L + 2 \cdot \pi \cdot (D/2)^2 = 2.294 \text{ sq. cm}$, 6 circles surface area = $2.294 \cdot 6 = 13.764 \text{ sq. cm}$
2265	
2272	straighten the chain and calculate
2289	Straighten one ring, calculate it's surface area as a cylinder, then multiply the amount of rings.
2290	
2293	Education used to calculated the surface $V = \pi \cdot r^2 \cdot h$. The volume of the chain was determined by displacement on water, then taken as cylinder surface
2295	
2296	Using Calipers and measured as a solid cylinder
2301	First we take 1 pcs of Chain and then measure the surface area as like Cylinder Surface area .
2303	The surface area of a link was measured using the formula $A = 2 \cdot \pi \cdot r \cdot l$. Total surface area = 6A
2309	Area of cylindrical & Area of circle; Area of one ring= 2.09 cm ² Area of six ring= 12.54 cm ²
2310	calculate the surface area of cylinder using vernier caliper
2311	The total surface area is the sum of six cylindrical chain.
2330	Assume is cylinder and calculate for cylinder area
2347	
2350	
2352	

lab	How was the surface of the chain measured and calculated?
2357	
2362	
2363	
2365	How to measured and calculated the surface of the object: Approximate the sample as a long cylinder, and measure the diameter and length, then calculate the surface using formula $\pi \cdot D \cdot \text{diameter} \cdot \text{length}$.
2366	Consider the sample as six cylinders , measure the length and diameter, calculate the surface area
2369	
2370	First take a plastic ring straightened to the cylinder to calculate the surface area, and then multiplied by six.
2375	
2379	
2380	We considered the object like a cylinder & the edge of the object as a circle
2385	Measurement of diameter and length of one segment. Multiplication by 6.
2390	Square, Small Circle & Rectangular Edges
2403	$S = \pi \cdot d(\text{average}) \cdot L \cdot 6 = 3.14 \cdot 0.178 \cdot 3.710 \cdot 6 \text{ cm}^2 = 12.45 \text{ cm}^2$
2410	
2429	
2432	
2442	We open the branch and take one part as a cylinder. Then finally multiply by six.
2459	$A = 2\pi r^2 + h(2\pi r)$; $h = 3.797 \text{ cm}$; $r = 0.0885 \text{ cm}$
2462	digital calliper
2475	link is similar as a cylinder with a surface of 1 link = $3.14 \cdot D \cdot L$. D=diameter, L=length. We have 6 links so we have multiplied the result of the surface by 6.
2489	Measured thickness in vernier caliper. Length was measured with thread marking.
2492	Separate the chain and make it straight for length measurement
2495	Archimedes' principle
2496	Remove a ring, then ring straightened, measured using vernier calipers.
2497	surface determination of one ring - then multiply by 6
2500	$S = d \cdot D \cdot L \cdot 6$ (S:Surface Area, d:diameter,L:length of one Oring)
2511	
2514	
2516	Used Digital caliper, Ruler and Tape measure.
2538	1 part is calculated as an ellipse: medium of inner and outer diameter = central line; the solid has an outer surface of a cylinder; tiny parts are flat, not round; these are calculated in difference to the round parts and the difference is added; the result is multiplied by 6 (6 parts of the chain)
2553	Draw the sample, divide to desired shapes and calculate the total surface
2560	It's consist 6 twisted ring, considered each as a whole cylinder. We used the law $2\delta r(r+h) \cdot 6$
2563	measured with digital vernier caliper; calculated as outside surface of a cylinder
2567	Each ring measure the length(h), then measure radius(r), using formula $2\pi r h \cdot 6$.
2590	Object was assimilated to a series of cylinders and circumferences. Area was measured with caliper.
2605	First measure the area of a circle, then multiplied by 6 to calculate the total area.
2613	Total Surface Area =Surface Area of one Ring x Total Number of Rings(6)
2624	
2629	
2653	measured the surface area of one link and then multiplied it with 6 for six links
2657	used by digimatic caliper
2666	we open one ring of chain and calculate the total surface of the cylinder (lateral+ 2 bases) and then we multiply the result for 6 rings
2674	
2678	The surface of the chain is calculated by multiplying the surface of a ring by 6. The surface of a ring is calculated by decomposing it into cylinders, rectangles and ellipses.
2705	The sample was elongated and considered as a cylinder. A second approach consists in considering the sample as an ellipsoidal torus. Both give as us a similar value.
2713	From cylinder area

lab	How was the surface of the chain measured and calculated?
2719	
2721	surface of a cylindre multiplicated with 6 (average of diameter, length with filament)
2737	The chain made up of several loops, the loop seems cylinder. The cylinder's diameter measure by vernier caliper, every loop measure five point and average them. Then copy the loop to the paper, using the filament simulate the shape of loop and calculated the length. Lastly, according to the formula calculate the cylinder area.
2741	The chain has 6 loops. One loop should be lengthened; it is a cylinder.
2743	Each ring of the plastic chain was considered as an ellipse. Its perimeter was measured together with its thickness. Then the surface of the associated cylinder was calculated and multiplied by the number of rings of the chain.
2758	
2766	we assumed it to be a cylinder and calculated diameter, we applied formula, "3.14* h*r^2" * 6 pc
2783	We used digital calipers to measure the length and diameter of each link, then added for total SA
2787	We unchain the sample and observe it like six cylinders. Calc.formula 6x (2xr2π+2rth).
3100	$S = \pi D L + 1/2 \pi D^2$, S:area, D:diameter, L:length
3110	
3116	Measured by calibrated digital caliper
3118	
3146	
3150	calculated as cylinder
3153	Geometric approximation
3154	
3160	$A_{total} = 2A_{circunferencia} + A_{cilindro} = 2\delta r^2 + 2\delta r(r+h)$
3172	3D scan
3176	-
3179	caliper was used.
3182	Using equation $2 \times 3.14 \times r \times h$ when r:radius of chain and h: length of each chain and times 6.
3183	One piece of the chain was stretched out and measured, the surface was presumed as cylinder. For the final result the area for the one piece measured was multiplied by 6.
3185	Based on the cylinder surface area calculation
3191	Use a micrometer to measure the diameter of the chain $d=1.72\text{mm}$, that is, the radius 0.86mm . Use the string to measure the length of the axle wire of ring body, the length is 38.62mm . Set the target samples as a bent cylinder to calculate the surface area, total surface area of the single ring is 2.14cm^2 , so the total surface area of six rings is 12.84cm^2 .
3197	We thought the sample as the combination of 6 cylindrical rings.
3200	regard this ring as an ellipse and use elliptical area formula to calculate it
3209	One chain Straightened out and it become a cylinder and than calculate a cylinder surface area.
3210	Sample= $6 \times (2 \times \text{cylinder} + \text{torus}) = 6 \times (2 \times 2\delta R h + 4\delta^2 R r)$
3214	Measured the length and width of a single metal ring plus the sectional area, calculated to 6 rings.
3218	
3220	By using thread and Vernier caliper
3225	Measure radius of inner and outer circle. Calculate as ring by using (sum of radius x Pi x 2 / 2)
3228	Separate one part from the chain and calculate surface area of the part following cylinder formula.
3233	We considered one cylinder + two circles
3237	One piece of the chain separated to 12 parts and surface of these parts are calculated as cylinder
8008	

APPENDIX 5

Number of participants per country

6 labs in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
2 labs in CAMBODIA, Kingdom of
6 labs in FRANCE
12 labs in GERMANY
1 lab in GUATEMALA
10 labs in HONG KONG
8 labs in INDIA
4 labs in INDONESIA
8 labs in ITALY
1 lab in JAPAN
4 labs in KOREA
1 lab in LUXEMBOURG
1 lab in MEXICO
1 lab in MOROCCO
29 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in SERBIA
1 lab in SINGAPORE
2 labs in SPAIN
1 lab in SRI LANKA
2 labs in SWITZERLAND
2 labs in TAIWAN R.O.C.
2 labs in THAILAND
2 labs in TUNISIA
6 labs in TURKEY
1 lab in U.S.A.
3 labs in UNITED KINGDOM
6 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	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request of participant
ex	= test result excluded from the statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported

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