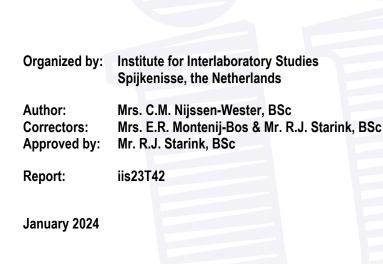


Institute for Interlaboratory Studies

> Results of Proficiency Test Heavy Metals by Perspiration in Textile November 2023



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1 INTRODUCTION

Since the 1990's many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for textiles some Ecolabelling schemes are imposing environmental requirements for textile products on a voluntary basis e.g. EU Ecolabel regulation 2014/350/EU, OEKO-TEX® Standard (Switzerland), bluesign® Restricted Substances List (Switzerland) and American Apparel and Footwear Association (United States).

Since 2002 the Institute of Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Heavy Metals by Perspiration in Textile every year. During the annual proficiency testing program 2023 it was decided to continue the proficiency test for the determination of Heavy Metals by Perspiration in Textile.

In this interlaboratory study 78 laboratories in 24 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of Heavy Metals by Perspiration in Textile 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 a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.

It was decided to send two different textile samples of approximately 3 grams each labelled #23740 and #23741 respectively.

The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. 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 first sample a batch of purple cotton pieces was selected which was artificially fortified with Copper by a third party. After homogenization 100 small plastic bags were filled with approximately 3 grams each and labelled #23740.

The batch for sample #23740 was used in a previous proficiency test on Heavy Metals by Perspiration in Textile as sample #14205 in iis14A03. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of green cotton pieces was selected which was artificially fortified with Chromium and Mercury by a third party. After homogenization 100 small plastic bags were filled with approximately 3 grams each and labelled #23741. The batch for sample #23741 was used in a previous proficiency test on Heavy Metals by Perspiration in Textile as sample #18631 in iis18A10. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories two textile samples labelled #23740 and #23741 respectively were sent on October 4, 2023.

2.5 ANALYZES

The participants were requested to determine on both samples: Antimony as Sb, Arsenic as As, Cadmium as Cd, Chromium as Cr, Cobalt as Co, Copper as Cu, Lead as Pb, Mercury as Hg and Nickel as Ni, applying the analysis procedure that is routinely used in the laboratory, but also to use preferably a solid/liquid ratio of 1/50 g/ml as prescribed in EN16711-2:15.

It was also requested to report if the laboratory was accredited for the requested components and to report some analytical details.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendices 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendices 1 and 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

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

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

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

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

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

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

3.2 GRAPHICS

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

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

3.3 Z-SCORES

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

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

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

The z-scores were calculated according to:

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples. Five participants reported test results after the final reporting date and five other participants did not report any test results. Not all participants were able to report all elements requested. In total 73 participants reported 196 numerical test results. Observed were 10 outlying test results, which is 5.1%. In proficiency tests 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 AND PER ELEMENT

In this section the reported test results are discussed per sample and per element. 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 5.

For the determination of Heavy Metals by Perspiration in Textile the EN16711-2 is considered to be the official test method. This method mentions the standard deviation and variation coefficient per element between laboratories. The reproducibility of each metal was calculated by multiplying the variation coefficient of the metal with 2.8.

sample #23740

<u>Copper as Cu</u>: The group of participants met the target requirements. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN16711-2:15.

The participants agreed on a concentration near or below the limit of detection for all other elements mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these elements. The reported test results are given in appendix 2.

sample #23741

<u>Chromium as Cr</u>: The group of participants met the target requirements. Three statistical outliers were observed. The calculated reproducibility after rejection of the outliers is in agreement with the requirements of EN16711-2:15.

<u>Mercury as Hg</u>: The group of participants met the target requirements. Four statistical outliers were observed. The calculated reproducibility after rejection of the outliers is in agreement with the requirements of EN16711-2:15.

The participants agreed on a concentration near or below the limit of detection for all other elements mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these elements. The reported test results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from the reference method are presented in the next two tables.

| Element | unit | n | average | 2.8 * sd | R(lit) |
|--------------|-------|----|---------|----------|--------|
| Copper as Cu | mg/kg | 67 | 55.9 | 13.1 | 25.1 |

Table 1: reproducibility of test on sample #23740

| Element | unit | n | average | 2.8 * sd | R(lit) |
|----------------|-------|----|---------|----------|--------|
| Chromium as Cr | mg/kg | 70 | 8.0 | 2.4 | 3.4 |
| Mercury as Hg | mg/kg | 49 | 0.20 | 0.13 | 0.17 |

Table 2: reproducibilities of tests on sample #23741

Without further statistical calculations it can be concluded that for all tests there is a good compliance of the group of participants with the reference test method.

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

| | November 2023 | November 2022 | November 2021 | November 2020 | November 2019 |
|------------------------------------|------------------|------------------|------------------|------------------|------------------|
| Number of reporting laboratories | 73 | 83 | 86 | 79 | 96 |
| Number of test results | 196 | 324 | 418 | 314 | 408 |
| Number of statistical outliers | 10 | 6 | 12 | 11 | 13 |
| Percentage of statistical outliers | 5.1% | 1.9% | 2.9% | 3.5% | 3.2% |

Table 3: comparison with previous proficiency tests

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

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTS, see next table.

| Element | November 2023 | November 2022 | November 2021 | November 2020 | 2010-2019 | EN16711-2 |
|----------------|------------------|------------------|------------------|------------------|-----------|-----------|
| Antimony as Sb | | 10% | | | 8-19% | 20% |
| Arsenic as As | | 8% | | | 9% | 20% |
| Cadmium as Cd | | | 9-12% | | 9-18% | 10% |
| Chromium as Cr | 10% | | | | 10-19% | 15% |
| Cobalt as Co | | | 12% | | 8-14% | 13% |
| Copper as Cu | 8% | 9% | 10% | 8-13% | 9-22% | 16% |
| Lead as Pb | | | | | 35-40% | 40% |
| Mercury as Hg | 24% | | | | 34-41% | 31% |
| Nickel as Ni | | 7% | 11% | 10% | 7-14% | 10% |

Table 4: development of the uncertainties over the years

The uncertainties observed in this PT are comparable or better than the uncertainties observed in previous PTs.

Sample #23740 was used in a previous PT as sample #14205 in iis14A03. The averages found in both PTs for this sample are similar. The calculated reproducibility for Chromium improved in the 2023 PT compared to the 2014 PT.

| Element | unit | sample #23740 | | | sample #14205 | | |
|--------------|-------|---------------|---------|---------|---------------|---------|---------|
| Liement | um | n | average | R(calc) | n | average | R(calc) |
| Copper as Cu | mg/kg | 67 | 55.9 | 13.1 | 81 | 57.05 | 16.69 |

Table 5: comparison of sample #23740 with #14205

Sample #23741 was used in a previous PT as sample #18634 in iis18A10. The averages found in both PTs for this sample are similar. The calculated reproducibility for the Chromium and Mercury improved in the 2023 PT compared to the 2018 PT.

| Element | unit | sample #23741 | | | sample #18631 | | |
|----------------|-------|---------------|---------|---------|---------------|---------|---------|
| Element | unit | n | average | R(calc) | n | average | R(calc) |
| Chromium as Cr | mg/kg | 70 | 8.0 | 2.4 | 88 | 9.1 | 2.5 |
| Mercury as Hg | mg/kg | 49 | 0.20 | 0.13 | 71 | 0.26 | 0.25 |

 Table 6: comparison of sample #23741 with #18631

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested and are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

- About 90% of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported elements.
- About 50% mentioned to use the sample as received and the other about 50% did further cut or further grind the sample prior to analysis.
- About 55% used a sample intake of 1 gram and about 35% used 0.5 grams.
- The vast majority of the participants used a ratio of 1 g to 50 mL. Remarkably, two

participants reported a ratio of 1g to 20-30 mL. Please note that in the method it is described that there can be a risk that not all the fabric is wetted sufficiently when a smaller amount of simulant is used.

For the elements present in the samples the calculated reproducibility is in agreement with the requirements of the target reproducibility, therefore no separate statistical analysis has been performed.

5 DISCUSSION

Almost all participants identified all added metals in both textile samples correctly: sample #23740 contained Copper and sample #23741 contained Chromium and Mercury.

When the results of this interlaboratory study were compared to the three guidelines from the table below, it was noticed that not all participants would have made identical decisions about the acceptability of sample #23740 and sample #23741.

All reporting participants except one would have rejected sample #23740 and all reporting participants except three would have rejected sample #23741.

| | OEKO-TEX® 100 Class 2: direct skin contact in mg/kg | bluesign® RSL Range A: next to skin use in mg/kg | 2014/350/EU: all textile products except baby in mg/kg |
|----------------|--|---|---|
| Method: | | EN16711-2 | EN ISO 105-E04 |
| Antimony as Sb | 30.0 | 10 | 30.0 |
| Arsenic as As | 1.0 | 0.2 | 1.0 |
| Cadmium as Cd | 0.1 | 0.1 | 0.1 |
| Chromium as Cr | 2.0 | 0.5 | 1.0 |
| Cobalt as Co | 4.0 | 1.0 | 1.0 |
| Copper as Cu | 50.0 | 50 | 50.0 |
| Lead as Pb | 1.0 | 1.0 | 1.0 |
| Mercury as Hg | 0.02 | 0.02 | 0.02 |
| Nickel as Ni | 4.0 | 1.0 | 1.0 |

Table 7: Overview from different ecolabelling schemes (latest version)

6 CONCLUSION

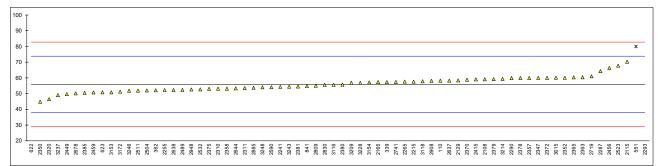
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

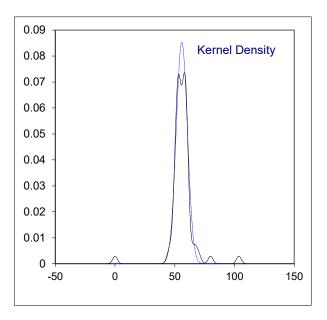
APPENDIX 1

Determination of Copper as Cu on sample #23740; results in mg/kg

| | | Copper as Cu on samp | | | |
|--------------|------------------------|----------------------|-----------|----------------|-------------------------|
| lab | method | value | mark | z(targ) | remarks |
| 110 | EN16711-2 | 58.23 | | 0.26 | |
| 210 | 10040 | | | | |
| 339 | ISO105E04 | 57.382 | | 0.16 | |
| 362 | In house | 52.1 | | -0.43 | |
| 551 622 | ISO105E04 | 79.9781 | R(0.01) | 2.69 | |
| 622 | EN16711-2 | 0.000 | R(0.01) | -6.25 | |
| 623 841 | EN16711-2 | 50.79 54 7 | | -0.57 -0.14 | |
| 841 2108 | ISO105E04 EN16711-2 | 54.7 59.07 | | -0.14 0.35 | |
| 2108 | EN16711-2 EN16711-2 | 70.29 | | 1.61 | |
| 2113 | EN16711-2 EN16711-2 | 58.3957 | | 0.28 | |
| 2129 | EN16711-2 EN16711-2 | 57.366 | | 0.20 | |
| 2215 | | 57.619 | | 0.10 | |
| 2241 | EN16711-2 | 54.27 | | -0.18 | |
| 2255 | EN16711-2 | 52.30 | | -0.40 | |
| 2265 | EN16711-2 | 57.6 | | 0.19 | |
| 2287 | EN16711-2 | 64.28 | | 0.93 | |
| 2290 | EN16711-2 | 59.91 | | 0.45 | |
| 2293 | EN16711-2 | 103.7 | C,R(0.01) | 5.34 | first reported: 1605.99 |
| 2310 | EN16711-2 | 53.1 | | -0.32 | |
| 2311 | EN16711-2 | 53.55 | | -0.27 | |
| 2320 | EN16711-2 | 46.428 | | -1.06 | |
| 2347 | EN16711-2 | 60.00 44.857 | | 0.46 | |
| 2350 2352 | EN16711-2 EN16711-2 | 44.857 60.1 | | -1.24 0.47 | |
| 2352 | EN16711-2 EN16711-2 | 59.96 | | 0.47 | |
| 2358 | EN16711-2 | 53.139 | | -0.31 | |
| 2363 | EN16711-2 | 60.35 | | 0.49 | |
| 2365 | EN16711-2 | 60.30 | | 0.49 | |
| 2370 | ISO105E04 | 58.8 | | 0.32 | |
| 2372 | EN16711-2 | 60 | | 0.46 | |
| 2375 | EN16711-2 | 53 | | -0.33 | |
| 2378 | EN16711-2 | 59.96 | | 0.45 | |
| 2379 | EN16711-2 | 59.2157 | | 0.37 | |
| 2380 | EN16711-2 | 55.743 | | -0.02 | |
| 2381 | EN16711-2 | 54.50 | | -0.16 | |
| 2385 | EN16711-2 | 50.5 | | -0.61 | |
| 2415 | EN16711-2 | 59.0 49.678 | | 0.34 | |
| 2449 2456 | ISO105E04 EN16711-2 | 49.678 66.34 | | -0.70 1.16 | |
| 2450 | EN16711-2 EN16711-2 | 50.76 | | -0.58 | |
| 2489 | EN16711-2 | 52.37 | | -0.30 | |
| 2504 | DIN54233-3 | 52.016 | | -0.44 | |
| 2511 | EN16711-2 | 51.9 | | -0.45 | |
| 2523 | | 67.747 | | 1.32 | |
| 2532 | EN16711-2 | 52.7 | | -0.36 | |
| 2561 | | | | | |
| 2582 | | | | | |
| 2590 | EN16711-2 | 54.06 | | -0.21 | |
| 2637 | EN16711-2 | 58.3 | | 0.27 | |
| 2638 | EN16711-2 | 52.31 53.34 | | -0.40 | |
| 2644 2678 | EN16711-2 EN16711-2 | 53.34 50.1 | | -0.29 -0.65 | |
| 2078 | LINIO7 11-2 | 61 | | -0.05 | |
| 2734 | | | | | |
| 2741 | EN16711-2 | 57.42 | | 0.17 | |
| 2758 | EN16711-2 | not determined | | | |
| 2809 | EN16711-2 | 55.0 | | -0.10 | |
| 2830 | EN16711-2 | 55.593 | | -0.04 | |
| 2885 | In house | 53.71 | | -0.25 | |
| 2908 | EN16711-2 | 58.09 | | 0.24 | |
| 2948 | EN16711-2 | 52.62 | | -0.37 | |
| 2989 | | | | | |
| 3015 | EN16711-2 | 60.06 | | 0.46 | |
| 3116 | EN16711-2 | 55.6 57.811 | C | -0.04 | first reported: 34,100 |
| 3118 3153 | EN16711-2 EN16711-2 | 57.811 50.799 | С | 0.21 -0.57 | first reported: 34.199 |
| 3153 | EN16711-2 EN16711-2 | 50.799 57.11 | | -0.57 0.13 | |
| 3172 | EN16711-2 | 51.225 | | -0.52 | |
| 3209 | EN16711-2 | 56.844 | | 0.10 | |
| 3210 | EN16711-2 | <57.6726 | | | |
| 3214 | EN16711-2 | 59.36 | | 0.38 | |
| 3228 | EN16711-2 | 56.844 | | 0.10 | |
| 3237 | EN16711-2 | 49.00 | | -0.77 | |
| 3243 | EN16711-2 | 54.4 | | -0.17 | |
| | | | | | |

| lab | method | value | mark | z(targ) | remarks |
|------|-----------------------|--------------|------------|---------|--------------------|
| 3246 | EN16711-2 | 51.82 | | -0.46 | |
| 3248 | ISO105E04 | 54 | С | -0.21 | first reported: 33 |
| 6191 | EN16711-2 | not analyzed | | | |
| | normality | ОК | | | |
| | n | 67 | | | |
| | outliers | 3 | | | |
| | mean (n) | 55.9214 | | | |
| | st.dev. (n) | 4.67389 | RSD = 8.4% | | |
| | R(calc.) | 13.0869 | | | |
| | st.dev.(EN16711-2:15) | 8.94742 | | | |
| | R(EN16711-2:15) | 25.0528 | | | |
| | . , | | | | |

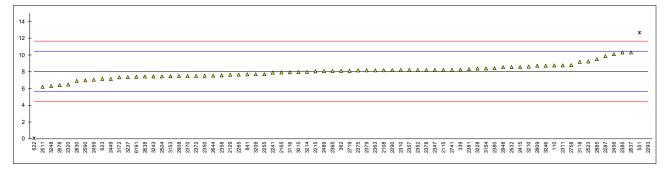


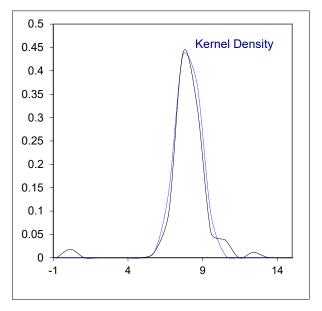


Determination of Chromium as Cr on sample #23741; results in mg/kg

| | | | | _// | |
|--------------|------------------------|----------------|-----------|------------------------|-----------------------|
| lab 110 | method | value | mark | z(targ) 0.58 | remarks |
| 110 210 | EN16711-2 | 8.736 | | 0.58 | |
| 339 | ISO105E04 | 8.263 | | 0.19 | |
| 362 | In house | 8.1 | | 0.06 | |
| 551 | ISO105E04 | 12.6574 | R(0.01) | 3.84 | |
| 622 | EN16711-2 | 0.054 | R(0.01) | -6.62 | |
| 623 841 | EN16711-2 | 7.13 7.68 | | -0.75 -0.29 | |
| 841 2108 | ISO105E04 | 7.68 8.164 | | -0.29 0.11 | |
| 2100 | EN16711-2 | 8.21 | | 0.15 | |
| 2129 | EN16711-2 | 7.6346 | | -0.33 | |
| 2165 | EN16711-2 | 7.916 | | -0.10 | |
| 2215 | | 8.028 | | 0.00 | |
| 2241 | EN16711-2 | 7.896 | | -0.11 | |
| 2255 2265 | EN16711-2 EN16711-2 | 7.70 7.65 | | -0.28 -0.32 | |
| 2203 | EN16711-2 | 9.856 | | 1.51 | |
| 2290 | EN16711-2 | 8.19 | | 0.13 | |
| 2293 | EN16711-2 | 238.2 | C,R(0.01) | 191.03 | first reported: 13.29 |
| 2310 | EN16711-2 | 8.2 | | 0.14 | |
| 2311 2320 | EN16711-2 EN16711-2 | 8.744 6.473 | | 0.59 -1.29 | |
| 2320 | EN16711-2 EN16711-2 | 8.21 | | -1.29 | |
| 2350 | EN16711-2 | 7.511 | | -0.43 | |
| 2352 | EN16711-2 | 8.2 | | 0.14 | |
| 2357 | EN16711-2 | 8.20 | | 0.14 | |
| 2358 | EN16711-2 | 7.561 | | -0.39 | |
| 2363 2365 | EN16711-2 EN16711-2 | 8.16 8.09 | | 0.11 0.05 | |
| 2305 | ISO105E04 | 7.49 | | -0.45 | |
| 2372 | EN16711-2 | 7.5 | | -0.44 | |
| 2375 | EN16711-2 | 8.15 | | 0.10 | |
| 2378 | EN16711-2 | 8.20 | | 0.14 | |
| 2379 | EN16711-2 | 8.1562 | | 0.10 | |
| 2380 2381 | EN16711-2 EN16711-2 | 8.413 8.30 | | 0.32 0.22 | |
| 2385 | EN16711-2 | 10.3 | | 1.88 | |
| 2415 | EN16711-2 | 8.56 | | 0.44 | |
| 2449 | | 7.13 | | -0.75 | |
| 2456 | EN16711-2 | 10.09 | | 1.71 | |
| 2459 2489 | EN16711-2 EN16711-2 | 7.05 8.08 | | -0.82 0.04 | |
| 2409 2504 | DIN54233-3 | 7.442 | | -0.49 | |
| 2511 | EN16711-2 | 6.2 | | -1.52 | |
| 2523 | | 9.219 | | 0.98 | |
| 2532 | EN16711-2 | 8.55 | | 0.43 | |
| 2561 2582 | | | | | |
| 2582 2590 | EN16711-2 | 6.97 | | -0.88 | |
| 2637 | EN16711-2 | 10.3 | | 1.88 | |
| 2638 | EN16711-2 | 7.43 | | -0.50 | |
| 2644 | EN16711-2 | 7.54 | | -0.41 | |
| 2678 | EN16711-2 | 6.4 8 1 | | -1.35 | |
| 2719 2734 | | 8.1 | | 0.06 | |
| 2734 | EN16711-2 | 8.222 | | 0.16 | |
| 2758 | EN16711-2 | 8.797 | | 0.63 | |
| 2809 | EN16711-2 | 8.70 | | 0.55 | |
| 2830 | EN16711-2 | 6.90 | | -0.94 | |
| 2885 2908 | EN16711-2 | 9.497 7.475 | | 1.22 -0.46 | |
| 2908 2948 | EN16711-2 EN16711-2 | 8.53 | | -0.46 | |
| 2989 | | | | | |
| 3015 | EN16711-2 | 7.96 | | -0.06 | |
| 3116 | EN16711-2 | 7.93 | | -0.09 | |
| 3118 | EN16711-2 | 9.179 | | 0.95 | |
| 3153 3154 | EN16711-2 EN16711-2 | 7.452 8.39 | | -0.48 0.30 | |
| 3154 | EN16711-2 EN16711-2 | 8.39 7.325 | | -0.59 | |
| 3209 | EN16711-2 | 7.684 | | -0.29 | |
| 3210 | EN16711-2 | 8.6153 | | 0.48 | |
| 3214 | EN16711-2 | 7.98 | | -0.04 | |
| 3228 | EN16711-2 | 8.376 | | 0.29 | |
| 3237 3243 | EN16711-2 EN16711-2 | 7.36 7.43 | | -0.56 -0.50 | |
| 5245 | | 7.45 | | -0.50 | |

| lab | method | value | mark | z(targ) | remarks |
|------|-----------------------|---------|-------------|---------|---------|
| 3246 | EN16711-2 | 8.72 | | 0.57 | |
| 3248 | ISO105E04 | 6.3 | | -1.44 | |
| 6191 | EN16711-2 | 7.386 | | -0.54 | |
| | normality | suspect | | | |
| | n | 70 | | | |
| | outliers | 3 | | | |
| | mean (n) | 8.0326 | | | |
| | st.dev. (n) | 0.84060 | RSD = 10.5% | | |
| | R(calc.) | 2.354 | | | |
| | st.dev.(EN16711-2:15) | 1.20489 | | | |
| | R(EN16711-2:15) | 3.3737 | | | |

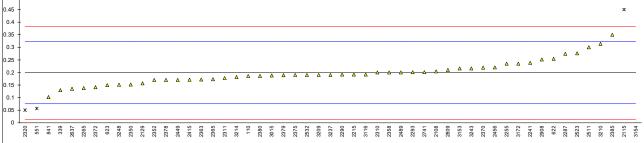


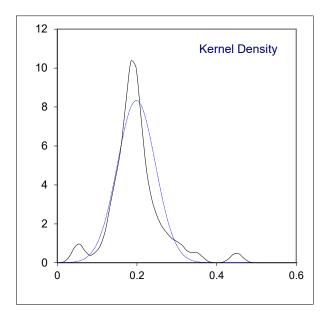


Determination of Mercury as Hg on sample #23741; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|--------------|------------------------|----------------|-----------|----------------|---------------------------------------|
| 110 | EN16711-2 | 0.186 | main | -0.21 | - Change |
| 210 | | 0.180 | | -0.21 | |
| 339 | ISO105E04 | 0.13 | | -1.12 | |
| 362 | In house | <0.01 | | <-3.06 | possibly a false negative test result |
| 551 | ISO105E04 | 0.0568 | R(0.01) | -2.30 | |
| 622 | EN16711-2 | 0.254 | · · · · | 0.90 | |
| 623 | EN16711-2 | 0.15 | | -0.79 | |
| 841 | ISO105E04 | 0.102 | С | -1.57 | first reported: 2.2 |
| 2108 | | 0.2043 | | 0.09 | |
| 2115 | EN16711-2 | 0.45 | R(0.01) | 4.08 | |
| 2129 | EN16711-2 | 0.1569 | | -0.68 | |
| 2165 2215 | EN16711-2 | <0.5 0.192 | | -0.11 | |
| 2213 | EN16711-2 | 0.238 | | 0.64 | |
| 2255 | EN16711-2 | 0.235 | | 0.59 | |
| 2265 | EN16711-2 | 0.138 | | -0.99 | |
| 2287 | EN16711-2 | 0.2742 | | 1.22 | |
| 2290 | EN16711-2 | 0.192 | | -0.11 | |
| 2293 | EN16711-2 | 0.201 | | 0.04 | |
| 2310 | EN16711-2 | 0.2 | | 0.02 | |
| 2311 | EN16711-2 | 0.178 | | -0.34 | |
| 2320 | EN16711-2 | 0.05 | R(0.01) | -2.41 | |
| 2347 | | | | | |
| 2350 | EN16711-2 | 0.152 | | -0.76 | |
| 2352 2357 | EN16711-2 | 0.17 | | -0.47 | |
| 2357 2358 | EN16711-2 | 0.2 | | 0.02 | |
| 2356 | EN16711-2 EN16711-2 | 0.2 | | -0.44 | |
| 2365 | EN16711-2 | 0.172 | | -0.42 | |
| 2370 | ISO105E04 | 0.219 | | 0.33 | |
| 2372 | EN16711-2 | 0.141 | | -0.94 | |
| 2375 | EN16711-2 | 0.19 | | -0.14 | |
| 2378 | EN16711-2 | 0.17 | | -0.47 | |
| 2379 | EN16711-2 | 0.1890 | | -0.16 | |
| 2380 | EN16711-2 | 0.186 | | -0.21 | |
| 2381 | | | | | |
| 2385 | EN16711-2 | 0.35 | | 2.45 | |
| 2415 2449 | EN16711-2 | 0.171 0.17 | | -0.45 -0.47 | |
| 2449 2456 | EN16711-2 | 0.22 | | -0.47 | |
| 2459 | EN16711-2 | ND | | | |
| 2489 | EN16711-2 | 0.2 | | 0.02 | |
| 2504 | DIN54233-3 | < 0.500 | | | |
| 2511 | EN16711-2 | 0.3 | | 1.64 | first reported 1.080 |
| 2523 | | 0.2763 | С | 1.26 | |
| 2532 | EN16711-2 | 0.19 | | -0.14 | |
| 2561 | | | | | |
| 2582 | | | | | |
| 2590 | EN16711-2 | < L.O.Q | | | |
| 2637 | EN16711-2 | 0.135 | | -1.04 | |
| 2638 | EN16711-2 | not analyzed | | | |
| 2644 | EN16711-2 | not detected | | | |
| 2678 2719 | EN16711-2 | not detected | | | |
| 2719 | | | | | |
| 2734 | EN16711-2 | 0.201 | | 0.04 | |
| 2758 | EN16711-2 | not determined | | | |
| 2809 | EN16711-2 | 0.210 | | 0.18 | |
| 2830 | | | | | |
| 2885 | | Not detected | | | |
| 2908 | EN16711-2 | 0.2518 | | 0.86 | |
| 2948 | | | | | |
| 2989 | | | | | |
| 3015 | EN16711-2 | 0.188 | | -0.18 | |
| 3116 | EN16711-2 | 0.192 | | -0.11 | nonsibly a false norstive test result |
| 3118 3153 | EN16711-2 EN16711-2 | <0.01 0.216 | | <-3.06 0.28 | possibly a false negative test result |
| 3153 | EN16711-2 | 3.24 | C,R(0.01) | 49.34 | first reported: 1.52 |
| 3154 | EN16711-2 | 0.235 | 0,1(0.01) | 49.34 0.59 | morroportou. 1.02 |
| 3209 | EN16711-2 | 0.190 | | -0.14 | |
| 3210 | EN16711-2 | 0.3137 | | 1.86 | |
| 3214 | EN16711-2 | 0.182 | | -0.27 | |
| 3228 | | | | | |
| 3237 | EN16711-2 | 0.19 | | -0.14 | |
| 3243 | EN16711-2 | 0.216 | | 0.28 | |
| | | | | | |

| 3246 EN16711-2 nd 3248 ISO105E04 0.151 -0.78 6191 EN16711-2 not analyzed normality suspect n 49 outliers 4 st.dev. (n) 0.04783 RSD = 24% R(calc.) 0.1339 st.dev.(EN16711-2:15) st.dev.(EN16711-2:15) 0.0726 | lab | method | value | mark | z(targ) | remarks |
|--|--------|-------------|--------------|-----------|---------|---------|
| 6191 EN16711-2 not analyzed normality suspect n 49 outliers 4 mean (n) 0.1988 st.dev. (n) 0.04783 RSD = 24% R(calc.) 0.1339 st.dev.(EN16711-2:15) st.dev. (EN16711-2:15) 0.06163 | 3246 | EN16711-2 | nd | | | |
| normality suspect n 49 outliers 4 mean (n) 0.1988 st.dev. (n) 0.04783 RSD = 24% R(calc.) 0.1339 st.dev.(EN16711-2:15) 0.06163 | 3248 | SO105E04 | 0.151 | | -0.78 | |
| n 49 outliers 4 mean (n) 0.1988 st.dev. (n) 0.04783 RSD = 24% R(calc.) 0.1339 st.dev. (EN16711-2:15) 0.06163 | 6191 I | EN16711-2 | not analyzed | | | |
| outliers 4 mean (n) 0.1988 st.dev. (n) 0.04783 RSD = 24% R(calc.) 0.1339 st.dev. (EN16711-2:15) 0.06163 | | normality | suspect | | | |
| mean (n)0.1988st.dev. (n)0.04783RSD = 24%R(calc.)0.1339st.dev. (EN16711-2:15)0.06163 | 1 | n | 49 | | | |
| st.dev. (n) 0.04783 RSD = 24% R(calc.) 0.1339 st.dev.(EN16711-2:15) 0.06163 | (| outliers | 4 | | | |
| R(calc.) 0.1339 st.dev.(EN16711-2:15) 0.06163 | 1 | mean (n) | 0.1988 | | | |
| st.dev.(EN16711-2:15) 0.06163 | 5 | st.dev. (n) | 0.04783 | RSD = 24% | | |
| st.dev.(EN16711-2:15) 0.06163 | | R(calc.) | 0.1339 | | | |
| | | | 0.06163 | | | |
| | | | 0.1726 | | | |
| |).5 т | | | | | |
| 5 | 5 | | | | | |





APPENDIX 2 Other reported elements

Other reported elements on sample #23740; results in mg/kg

| lab | Sb | As | Cd | Cr |
|--------------|----------------------------|----------------------------|----------------------------|----------------------------|
| 110 | not detected, <0.1 mg/kg |
| 210 | | | | |
| | not detected | not detected | not detected | not detected |
| | <1.0 | <0.1 | <0.05 | <0.05 |
| 551 | | 0.0069 | 0.0000 | 0.0017 |
| | 0.036 Not Detected | 0.000 Not Detected | 0.000 Not Detected | 0.098 Not Detected |
| 623 841 | Not Detected <0.2 | Not Detected <0.2 | Not Detected <0.2 | Not Detected <0.2 |
| 2108 | <0.2 not detected | <0.2 not detected | <0.2 not detected | <0.2 not detected |
| 2100 | | | | |
| 2115 | not detected | not detected | not detected | not detected |
| 2165 | | Not Detected | Not Detected | Not Detected |
| 2215 | | <0.02 | <0.02 | <0.1 |
| 2241 | | not applicable | not applicable | not applicable |
| 2255 | | Not Detected | Not Detected | Not Detected |
| 2265 | | not detected | not detected | not detected |
| 2287 | | <0.2 | <0.1 | <1.0 |
| 2290 | | <0.1 | <0.03 | <0.5 |
| 2293 | | 0.229 | 0.0019 | 0.29 |
| | not detected | not detected | not detected | not detected |
| 2311 | Not Detected | Not Detected | Not Detected | Not Detected |
| 2320 | <3 | <0.06 | <0.03 | <0.3 |
| 2347 | <1 | <0.1 | <0.05 | <1 |
| 2350 | <0.5 | <0.02 | <0.02 | <0.1 |
| 2352 | | | | |
| 2357 | | <0.1 | <0.05 | <0.5 |
| | not detected | not detected | not detected | not detected |
| 2363 | | not detected | not detected | not detected |
| 2365 | | <0.06 | <0.03 | <0.3 |
| 2370 | | <0.2 | <0.1 | <0.5 |
| 2372 | | <0.2 | <0.1 | <0.5 |
| 2375 | | < 0.06 | < 0.03 | <0.3 |
| 2378 | | <0.06 | <0.03 | <0.3 |
| 2379 | | Not detected | Not detected | Not detected |
| 2380 | | | | |
| 2381 | | | | |
| 2385 | | <0.1 | <0.05 | <0.1 |
| 2415 | | | | |
| 2449 | not detected | not detected | not detected | not detected |
| 2450 | | ND | ND | ND |
| 2439 | | Not Detected | Not Detected | Not Detected |
| | < 0.5000 | < 0.200 | < 0.200 | < 0.200 |
| 2511 | | | | |
| 2523 | | N.D. | N.D. | N.D. |
| | Not detected | Not detected | Not detected | Not detected |
| 2561 | | | | |
| 2582 | | | | |
| | < L.OQ | < L.OQ | < L.OQ | < L.OQ |
| | <0.005 | <0.01 | <0.005 | <0.01 |
| | not detected | not detected | not detected | not detected |
| | not detected | not detected | not detected | not detected |
| 2678 | not detected | not detected | not detected | not detected |
| 2719 | | | | |
| 2734 | | | | |
| 2741 | <3 | <0.1 | <0.05 | <0.5 |
| 2758 | not determined | not detected | not detected | not detected |
| | not detected | not detected | not detected | not detected |
| 2830 | | | | |
| | Not detected | Not detected | Not detected | Not detected |
| | Below quantification limit | Below quantification limit | Below quantification limit | Below quantification limit |
| 2948 | | | | |
| 2989 | | | | |
| 3015 | | <0.1 | < 0.03 | <0.5 |
| 3116 | | <0.02 | <0.02 | <0.1 |
| | <0.25 | <0.05 | <0.05 | <0.50 |
| 3153 | | < 0.3 | < 0.03 | < 0.5 |
| 3154 | not detected | not detected | not detected | not detected |
| | < 11.2 | < 0.02 | < 0.02 | < 0.1 |
| 3172 | | | | |
| 3172 3209 | <1.00 | <0.10 | <0.05 | <0.50 |
| 3172 | <1.00 <5 | | <0.05 <0.1 <0.03 | <0.50 <1 <0.5 |

| lab | Sb | As | Cd | Cr |
|------|--------------|--------------|--------------|--------------|
| 3228 | <0.5 | <0.02 | <0.02 | <0.5 |
| 3237 | | | | |
| 3243 | 0.008 | 0.022 | 0.008 | 0.015 |
| 3246 | nd | nd | nd | nd |
| 3248 | <0.5 | <0.02 | <0.02 | <0.1 |
| 6191 | not analyzed | not detected | not detected | not detected |

Other reported elements on sample #23740; results in mg/kg (continued)

| | Co | Pb | Hg | Ni |
|--------------|------------------------------|------------------------------|--------------------------|----------------------------|
| 210 | not detected, <0.1 mg/kg | not detected, <0.1 mg/kg | not detected, <0.1 mg/kg | 0.118 |
| | not detected | not detected | not detected | not detected |
| | <0.5 | <0.1 | <0.01 | <0.5 |
| 551 | 0.0023 | 0.0000 | 0.0000 | 0.0938 |
| 622 | 0.000 | 0.000 | 0.019 | 0.168 |
| | Not Detected | Not Detected | Not Detected | 0.1 |
| 841 | | < 0.2 | <0.1 | <0.2 |
| 2108 | not detected | not detected | not detected | 0.1626 0.075 |
| | not detected | not detected | not detected | not detected |
| | Not Detected | Not Detected | Not Detected | Not Detected |
| 2215 | | <0.1 | <0.02 | <0.2 |
| 2241 | not applicable | not applicable | not applicable | not applicable |
| | Not Detected | Not Detected | Not Detected | Not Detected |
| | not detected | not detected | not detected | not detected |
| | <1.0 <0.3 | <0.2 <0.1 | <0.02 <0.01 | <0.5 <0.3 |
| | 0.013 | 0 | 0.0 | 0.184 |
| | not detected | not detected | not detected | 0.11 |
| | Not Detected | Not Detected | Not Detected | 0.094 |
| 2320 | | <0.06 | <0.01 | <0.1 |
| | <0.5 | <0.1 | <0.01 | <0.5 |
| 2350 | | <0.06 | <0.005 | <0.1 |
| 2352 2357 | | <0.1 | <0.01 | <0.5 |
| | not detected | not detected | not detected | <0.5 not detected |
| | not detected | not detected | not detected | not detected |
| | <0.3 | <0.06 | <0.02 | <0.1 |
| 2370 | <0.1 | <0.2 | <0.02 | <0.5 |
| 2372 | | <0.2 | <0.02 | <0.5 |
| | <0.3 | <0.06 | <0.006 | <0.3 |
| | < 0.3 | <0.06 | <0.006 | <0.1 |
| 2379 | Not detected | Not detected | Not detected | 0.1297 |
| 2381 | | | | |
| 2385 | | <0.1 | <0.01 | <0.5 |
| 2415 | | | | |
| 2449 | | | | |
| | not detected | not detected | not detected | not detected |
| 2459 | | ND Net Detected | ND Not Detected | ND |
| | Not Detected < 0.200 | Not Detected < 0.200 | Not Detected < 0.500 | < 0.200 |
| 2511 | | | | |
| | N.D. | N.D. | N.D. | 0.095 |
| 2532 | Not detected | Not detected | Not detected | Not detected |
| 2561 | | | | |
| 2582 | | | | |
| | < L.OQ | < L.OQ | < L.OQ | < L.OQ |
| | <0.002 not detected | <0.005 not detected | <0.005 not analyzed | 0.12 not detected |
| | not detected | not detected | not detected | not detected |
| | not detected | not detected | not detected | not detected |
| 2719 | | | | |
| 2734 | | | | |
| 2741 | | <0.1 | <0.02 | <0.2 |
| | not detected | not detected | not determined | 0.142 not detected |
| 2809 | not detected | not detected | not detected | not detected |
| | Not detected | Not detected | Not detected | Not detected |
| | Below quantification limit | Below quantification limit | | Below quantification limit |
| 2948 | | | | |
| 2989 | | | | |
| 3015 | | <0.1 | <0.01 | <0.3 |
| 3116 | | <0.1 | <0.005 | 0.130 |
| 3118 3153 | <0.25 < 1 | <0.05 < 0.3 | <0.01 < 0.01 | <0.25 0.144 |
| | not detected | < 0.5 not detected | not detected | not detected |
| | < 0.1 | < 0.1 | < 0.005 | < 0.1 |
| | <0.50 | <0.10 | <0.02 | <0.50 |
| 3210 | | <0.2 | <0.02 | <1 |
| | <0.3 | <0.1 | <0.01 | <0.3 |
| | <0.5 | <0.02 | <0.02 | <0.5 |
| 3237 | | | | |
| 3243 | 0.008 | 0.038 | 0.003 | 0.16 |

| lab | Со | Pb | Hg | Ni |
|------|--------------|--------------|--------------|--------------|
| 3246 | nd | nd | nd | nd |
| 3248 | <0.1 | <0.1 | <0.005 | <0.01 |
| 6191 | not analyzed | not detected | not analyzed | not analyzed |

Other reported metals on sample #23741; results in mg/kg

| lab | Sb | As | Cd | Со |
|--------------|------------------------------|------------------------------|------------------------------|------------------------------|
| 110 | not detected, <0.1 mg/kg |
| 210 339 | not detected | not detected | not detected | not detected |
| | <1.0 | <0.1 | <0.05 | <0.5 |
| 551 | | 0.0088 | 0.0000 | 0.0047 |
| | 0.005 | 0.003 | 0.000 | 0.025 |
| 623 841 | Not Detected <0.2 | Not Detected <0.2 | Not Detected <0.2 | Not Detected ND |
| 2108 | not detected | not detected | not detected | not detected |
| 2115 | | | | |
| 2129 | not detected | not detected | not detected | not detected |
| 2165 2215 | Not Detected <0.5 | Not Detected <0.02 | Not Detected <0.02 | Not Detected <0.1 |
| 2241 | not applicable | not applicable | not applicable | not applicable |
| 2255 | Not Detected | Not Detected | Not Detected | Not Detected |
| 2265 | not detected | not detected | not detected | not detected |
| 2287 | <3.0 <1.0 | <0.2 <0.1 | <0.1 <0.03 | <1.0 <0.3 |
| 2293 | 0.306 | 0.16 | 0.0 | 0.013 |
| 2310 | not detected | not detected | not detected | not detected |
| 2311 | Not Detected | Not Detected | Not Detected | Not Detected |
| 2320 2347 | <3 <1 | <0.06 <0.1 | <0.03 <0.05 | <0.3 <0.5 |
| 2347 | | <0.02 | <0.03 | <0.3 |
| 2352 | | | | |
| | <1.0 | < 0.1 | <0.05 | <0.5 |
| 2358 2363 | not detected not detected | not detected not detected | not detected not detected | not detected not detected |
| 2365 | <1.0 | <0.06 | <0.03 | <0.3 |
| 2370 | | <0.2 | <0.1 | <0.1 |
| | <1.0 | <0.2 | <0.1 | <0.1 |
| 2375 2378 | <3 <3 | <0.06 <0.06 | <0.03 <0.03 | <0.3 <0.3 |
| 2379 | Not detected | Not detected | Not detected | Not detected |
| 2380 | | | | |
| 2381 | | | | |
| 2385 2415 | <0.1 | <0.1 | <0.05 | <0.1 |
| 2449 | | | | |
| 2456 | | not detected | not detected | not detected |
| 2459 | ND Nat Data at a d | ND Not Data at a d | ND Not Data at a | ND Not Detected |
| 2489 2504 | Not Detected < 0.500 | Not Detected < 0.200 | Not Detected < 0.200 | Not Detected < 0.200 |
| 2511 | | | | |
| 2523 | N.D. | N.D. | N.D. | N.D. |
| 2532 2561 | Not detected | Not detected | Not detected | Not detected |
| 2561 | | | | |
| | < L.OQ | < L.OQ | < L.OQ | < L.OQ |
| 2637 | 0.005 | <0.01 | <0.005 | 0.005 |
| | not detected not detected | not detected not detected | not detected not detected | not detected |
| | not detected | not detected | not detected | not detected not detected |
| 2719 | | | | |
| 2734 | | | | |
| 2741 2758 | <3 not determined | <0.1 not detected | <0.05 not detected | <0.5 not detected |
| | not detected | not detected | not detected | not detected |
| 2830 | | | | |
| | Not detected | Not detected | Not detected | Not detected |
| 2908 2948 | • | Below quantification limit | Below quantification limit | Below quantification limit |
| 2948 | | | | |
| 3015 | <1.0 | <0.1 | <0.03 | <0.3 |
| | <0.5 | < 0.02 | <0.02 | <0.1 |
| 3118 3153 | <0.25 < 1 | <0.05 < 0.3 | <0.05 < 0.03 | <0.25 < 1 |
| | not detected | not detected | not detected | not detected |
| | < 0.2 | < 0.02 | < 0.02 | < 0.1 |
| | <1.00 | <0.10 | <0.05 | <0.50 |
| 3210 3214 | <5 <1.0 | <0.2 <0.1 | <0.1 <0.03 | <1 <0.3 |
| | <0.5 | <0.02 | <0.03 | <0.3 |
| 3237 | | | | |
| 3243 | 0.063 | 0.022 | 0.005 | 0.004 |
| | | | | |

| lab | Sb | As | Cd | Со |
|------|--------------|--------------|--------------|--------------|
| 3246 | nd | nd | nd | nd |
| 3248 | <0.5 | <0.02 | <0.02 | <0.1 |
| 6191 | not analyzed | not detected | not detected | not analyzed |

Other reported metals on sample #23741; results in mg/kg (continued)

| | Cu | Pb | Ni |
|--------------|----------------------------|----------------------------|----------------------------|
| | 0.323 | not detected, <0.1 mg/kg | 0.175 |
| | | | |
| | not detected | not detected | not detected |
| | <5.0 | <0.1 | <0.5 |
| | 0.2155 | 0.0000 | 0.1208 |
| | 0.000 | 0.000 | 0.059 |
| | Not Detected | Not Detected | 0.14 |
| 841 | | <0.2 | <0.2 |
| | not detected | not detected | 0.2209 |
| 2115 | | | 0.15 |
| | not detected | not detected | not detected |
| | Not Detected | Not Detected | Not Detected |
| 2215 | so not applicable | <0.1 not applicable | <0.2 not applicable |
| | Not Detected | Not Detected | Not Detected |
| | not detected | not detected | not detected |
| 2203 | | <0.2 | <0.5 |
| 2290 | | <0.2 | <0.3 |
| | 37.06 C | 0.008 | 0.227 |
| | not detected | not detected | 0.13 |
| 2310 | | Not Detected | 0.095 |
| 2320 | | <0.06 | <0.1 |
| 2347 | - | <0.1 | <0.5 |
| 2350 | | <0.06 | 0.127 |
| 2352 | | | |
| 2357 | | <0.1 | <0.5 |
| 2358 | not detected | not detected | not detected |
| | not detected | not detected | not detected |
| 2365 | | <0.06 | 0.143 |
| 2370 | <5 | <0.2 | <0.5 |
| 2372 | <5 | <0.2 | <0.5 |
| 2375 | <5.0 | <0.06 | <0.3 |
| 2378 | <5.0 | <0.06 | <0.1 |
| 2379 | Not detected | Not detected | 0.1661 |
| 2380 | | | |
| 2381 | | | |
| 2385 | | <0.1 | <0.5 |
| 2415 | | | |
| 2449 | | | |
| | not detected | not detected | not detected |
| 2459 | | ND | ND |
| | Not Detected | Not Detected | 0.13 |
| | < 0.500 | < 0.200 | 0.200 |
| 2511 | | | |
| | 0.423 | N.D. | 0.175 |
| | Not detected | Not detected | 0.12 |
| 2561 | | | |
| 2582 | | | |
| 2590 2637 | < L.OQ 0.23 | < L.OQ 0.007 | < L.OQ 0.19 |
| | not detected | not detected | not detected |
| 2638 | | not detected | not detected |
| 2678 | | not detected | not detected |
| 2719 | | | |
| 2734 | | | |
| 2741 | | <0.1 | <0.2 |
| | not determined | not detected | 0.179 |
| | not detected | not detected | not detected |
| 2830 | | | |
| | Not detected | Not detected | 0.170 |
| 2908 | Below quantification limit | Below quantification limit | Below quantification limit |
| 2948 | | | |
| 2989 | | | |
| 3015 | | <0.1 | <0.3 |
| 3116 | | <0.1 | 0.163 |
| | <0.25 | <0.05 | <0.25 |
| 3153 | | < 0.3 | 0.122 |
| | not detected | not detected | not detected |
| 3172 | | < 0.1 | < 0.1 |
| | <1.00 | <0.10 | <0.50 |
| 3210 | | <0.2 | <1 |
| 3214 | | <0.1 | <0.3 |
| 3228 | | <0.02 | <0.5 |
| 3237 | | | |
| 3243 | 0.61 | 0.024 | 0.147 |
| | | | |

| lab | Cu | Pb | Ni |
|------|--------------|--------------|--------------|
| 3246 | nd | nd | nd |
| 3248 | <5 | <0.1 | <0.01 |
| 6191 | not analyzed | not detected | not analyzed |

Lab 2293 first reported: 107.70

APPENDIX 3 Analytical Details

| | 100/1501500 | | | |
|--------------|-------------|--------------------------------------|-------------------|--|
| lab | | sample preparation | Sample intake (g) | Ratio gram textile per ml |
| | Yes | Further cut | 1 g | 1 gram textile per 50 mL perspiration liquid |
| 210 | | | 1 ~ | 1 grom toxtile per E0 ml. pereniration liquid |
| | No Yes | Used as received Used as received | 1g | 1 gram textile per 50 mL perspiration liquid 1 gram textile per 50 mL perspiration liquid |
| 551 | | | 1g | i gram textile per 50 mL perspiration liquid |
| | | | 0.5.75 | 1 grom toytile par E0 ml. peropiration liquid |
| | Yes Yes | Used as received | 0.5 grams 0.5 | 1 gram textile per 50 mL perspiration liquid 1 gram textile per 50 mL perspiration liquid |
| | | Further cut | | |
| | Yes | Further cut Further cut | 0.5 grams | 1 gram textile per 50 mL perspiration liquid 1 gram textile per 50 mL perspiration liquid |
| 2108 2115 | | | 0,5 g | |
| 2115 | | Used as received Further cut | 1 g | 1 gram textile per 50 mL perspiration liquid |
| 2129 | | | 1g | 1 gram textile per 50 mL perspiration liquid 1 gram textile per 50 mL perspiration liquid |
| 2105 | | Used as received | 0.5g 1.0097 | 1 gram textile per 50 mL perspiration liquid |
| 2215 | | Further cut | 1.0097 | |
| 2241 | | Used as received | 0 0001/1 125 | 1 gram textile per 50 mL perspiration liquid |
| | | Used as received | 0.9001/1.125 | 1 gram textile per 50 mL perspiration liquid |
| 2265 | | Used as received | 1 gram | 1 gram textile per 30 mL perspiration liquid |
| 2287 | | Further cut | 0.5g | 1 gram textile per 50 mL perspiration liquid |
| 2290 | | | 1.0 | A many tartile and 50 miles and institution limited |
| 2293 | | Further cut | 1.0 grams | 1 gram textile per 50 mL perspiration liquid |
| 2310 | | Further cut | 4 | 1 gram textile per 50 mL perspiration liquid |
| 2311 | | Further cut | 1 | 1 gram textile per 50 mL perspiration liquid |
| 2320 | | Used as received | 0.5 | 0.5 gram textile per 25 mL perspiration liquid |
| 2347 | | | 0.5g | 1 gram textile per 50 mL perspiration liquid |
| 2350 | | Further cut | approx. 1.0g | 1 gram textile per 50 mL perspiration liquid |
| 2352 | | Used as received | 1g | 1 gram textile per 50 mL perspiration liquid |
| 2357 | | | | |
| 2358 | Yes | Further cut | 1g | 1 gram textile per 50 mL perspiration liquid |
| 2363 | | Used as received | 1g | 1 gram textile per 50 mL perspiration liquid |
| 2365 | Yes | Further cut | 0.5g | 0.5 gram textile per 25mL perspiration liquid |
| 2370 | Yes | Further cut | 1g | 1 gram textile per 50 mL perspiration liquid |
| 2372 | Yes | Further cut | 1g | 1 gram textile per 50 mL perspiration liquid |
| 2375 | Yes | Further cut | 0.5 gram | 1 gram textile per 50 mL perspiration liquid |
| 2378 | Yes | Used as received | 1g | 1 gram textile per 50 mL perspiration liquid |
| 2379 | Yes | Used as received | 0.5 g | 1 gram textile per 50 mL perspiration liquid |
| 2380 | | Further cut | 0.50 g | 0.50 g textile per 25 ml perspiration liquid |
| 2381 | | Used as received | 1g | 1 gram textile per 50 mL perspiration liquid |
| 2385 | | Used as received | -9 | 1 gram textile per 50 mL perspiration liquid |
| 2415 | | Used as received | 0.5 gram | 1 gram textile per 50 mL perspiration liquid |
| 2449 | | | ele gram | |
| 2456 | | Further cut | | 1 gram textile per 50 mL perspiration liquid |
| 2459 | | Used as received | 1 gram | 1 gram textile per 50 mL perspiration liquid |
| 2489 | | Further cut | 0.5015g/0.5006g | 1 gram textile per 50 mL perspiration liquid |
| 2504 | | #23740 : Further cut | 1 gram | 1 gram textile per 50 mL perspiration liquid |
| 2004 | 165 | #23741: used as recieved | rgiani | r grann texture per 50 mil perspiration liquid |
| 2511 | | #23741. used as recieved | | |
| 2523 | Voc | Used as received | 1.0000g | 1 gram textile per 50 mL perspiration liquid |
| | | Used as received | 1.0000g | |
| 2532 | | | | |
| 2561 | | | | |
| 2582 | | | 1 ~ | 1 grom toutile nor E0 rel generation limit |
| 2590 | | Used as received | 1g | 1 gram textile per 50 mL perspiration liquid |
| 2637 | | Used as received | 4 | 1 gram textile per 50 mL perspiration liquid |
| 2638 | | Further cut | 1 gm | 1 gram textile per 50 mL perspiration liquid |
| 2644 | | Used as received | 0.5 | 1 gram textile per 20 mL perspiration liquid |
| 2678 | | Used as received | 0.5 | 1 gram textile per 50 mL perspiration liquid |
| 2719 | | Further cut | 1g | 1 gram textile per 30 mL perspiration liquid |
| 2734 | | | | |
| 2741 | | Further cut | 1 | 1 gram textile per 50 mL perspiration liquid |
| 2758 | | Used as received | 1 | 1 gram textile per 50 mL perspiration liquid |
| 2809 | | Further cut | 1 | 1 gram textile per 50 mL perspiration liquid |
| 2830 | | Used as received | | 1 gram textile per 50 mL perspiration liquid |
| 2885 | | Further cut | 1.0 g | 1 gram textile per 50 mL perspiration liquid |
| 2908 | | Further cut | 1g | 1 gram textile per 50 mL perspiration liquid |
| 2948 | | | 1.0 | |
| 2989 | | | | |
| 3015 | | | | |
| 3116 | Yes | Further cut | 1g | 1 gram textile per 50 mL perspiration liquid |
| 3118 | | Used as received | 0.5 | 1 gram textile per 50 mL perspiration liquid |
| 3153 | | Further cut | 3 grams | 0.5 grams textile per 25ml perspiration Liquid |
| 3154 | | Further cut | 0.5 gram | 1 gram textile per 50 mL perspiration liquid |
| 3172 | | Used as received | 1 | 1 gram textile per 50 mL perspiration liquid |
| 3209 | | | | |
| 3209 | | Used as received | | 1 gram textile per 50 mL perspiration liquid |
| 3210 | | Further cut | 1 | 1 gram textile per 50 mL perspiration liquid |
| 3214 | | | 1 0.5g | |
| | | Used as received | | 1 gram textile per 50 mL perspiration liquid |
| 3237 | res | Used as received | 0.5g | 1 gram textile per 50 mL perspiration liquid |
| | | | | |

| lab | ISO/IEC1702 accr. | sample preparation | Sample intake (g) | Ratio gram textile per ml |
|------|-------------------|--------------------|-------------------|--|
| 3243 | Yes | Used as received | 0,5 | 1 gram textile per 50 mL perspiration liquid |
| 3246 | Yes | Further cut | 1,0 g | 1 gram textile per 50 mL perspiration liquid |
| 3248 | Yes | Used as received | 0.5G | 1 gram textile per 50 mL perspiration liquid |
| 6191 | Yes | Used as received | 1 | 1 gram textile per 50 mL perspiration liquid |

APPENDIX 4

Number of participants per country

3 labs in BANGLADESH

1 lab in BRAZIL

1 lab in BULGARIA 3 labs in FRANCE

7 labs in GERMANY

1 lab in GUATEMALA

4 labs in HONG KONG

4 labs in INDIA

3 labs in INDONESIA

6 labs in ITALY

1 lab in JAPAN

2 labs in KOREA, Republic of

3 labs in MOROCCO

12 labs in P.R. of CHINA

5 labs in PAKISTAN

2 labs in SRI LANKA

1 lab in SWITZERLAND

4 labs in TAIWAN

2 labs in THAILAND

2 labs in TUNISIA

2 labs in TURKEY

1 lab in U.S.A.

1 lab in UNITED KINGDOM

7 labs in VIETNAM

APPENDIX 5

Abbreviations

| С | = 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

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