Institute for
Interlaboratory Studies

# Results of Proficiency Test
# Total Metals in Metal/Metal Alloy
# June 2023


**Organized by:** Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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# 1 INTRODUCTION

Heavy metals can be found in everything from jewelry and watch cases to electronic components. While Lead, Cadmium, Mercury and other heavy metals serve a purpose they are highly toxic to humans. Nickel is one of the most abundant metallic elements, likely to be found in most metals and metal alloys in trace quantities, including coins. Lead is a heavy metal that has often been used in jewelry, to make the article heavier, brighten colors and to stabilize or soften plastic. But Lead does not break down in the environment and accumulates in the human body.
Cadmium is also a heavy metal that has been used for over a century in both fashion and fine jewelry products. Small amounts of Cadmium may be added to metal alloys to impart specific technical and functional attributes to the metal alloys. It may be present in jewelry as part of the metal alloy, solder or gold coating for electroforming / electroplating, or as a pigment or stabilizer in non-metal components.
The legislation covering the restrictions on metals is found in Regulation (EC) No 1907/2006 concerning the Registration, Evaluation, Authorization and Restriction of Chemicals (REACH). Presence of Lead has been limited in the Consumer Product Safety Improvement Act (CPSIA) of 2008. The limit of Lead is 100 mg/kg. In REACH there are limits mentioned for Cadmium of 100 mg/kg and for Lead 500 mg/kg.

Since 2021 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Metals in Metal/Metal Alloy every year. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of Metals in Metal/Metal Alloy.

In this interlaboratory study 53 laboratories in 19 countries registered for participation, see appendix 5 for the number of participants per country. In this report the results of the Metals in Metal/Metal Alloy proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

# 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one metal bracelet labelled #23630.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

## 2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant’s data. Feedback from the participants on the reported data is encouraged and customer’s satisfaction is measured on regular basis by sending out questionnaires.

## 2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

## 2.3 CONFIDENTIALITY STATEMENT

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

## 2.4 SAMPLES

A batch of bracelets was purchased from the local market. Each bracelet was packed separately and labelled #23630. The bracelet could be divided into two parts (see picture):


Part 1: decorated leaves at ends of the bracelet, further referred to as #23630-1.
Part 2: main part of the bracelet, further referred to as #23630-2.
The green line indicates the cutting line where the parts can be separated from each other.

The homogeneity of #23630-1 was checked by determination of Cadmium as Cd according to an in house method on 8 stratified randomly selected subsamples.

| | Cadmium as Cd in mg/kg |
|---|---|
| sample #23630-1 sub-1 | 24620 |
| sample #23630-1 sub-2 | 22595 |
| sample #23630-1 sub-3 | 23934 |
| sample #23630-1 sub-4 | 23161 |
| sample #23630-1 sub-5 | 24401 |
| sample #23630-1 sub-6 | 22581 |
| sample #23630-1 sub-7 | 22153 |
| sample #23630-1 sub-8 | 26355 |

Table 1: homogeneity test results of subsamples #23630-1

From the above test results the relative standard deviation (RSD) was calculated and compared with 0.3 times the average relative standard deviation obtained from twenty-four elements which were evaluated in the previous two iis PTs Total Metals in Metal/Metal Alloy in agreement with the procedure of ISO13528, Annex B2 in the next table.

| | Cadmium as Cd |
|---|---|
| RSD% observed | 5.9 |
| reference method | iis PTs |
| 0.3 x RSD% (reference method) | 6.6 |

Table 2: evaluation of the relative standard deviation of subsamples #23630-1

The calculated relative standard deviation is in agreement with 0.3 times the average relative standard deviation obtained from the evaluation of the elements from the previous two PTs. Therefore, homogeneity #23630-1 was assumed.

The homogeneity of #23630-2 was checked by determination of Cobalt according to an in house method on 7 stratified randomly selected subsamples.

| | Cobalt as Co<br>in mg/kg |
|---|---|
| sample #23630-2 sub-1 | 29.59 |
| sample #23630-2 sub-2 | 25.74 |
| sample #23630-2 sub-3 | 25.99 |
| sample #23630-2 sub-4 | 25.93 |
| sample #23630-2 sub-5 | 25.95 |
| sample #23630-2 sub-6 | 26.28 |
| sample #23630-2 sub-7 | 26.75 |

Table 3: homogeneity test results of subsamples #23630-2

From the above test results the relative standard deviation (RSD) was calculated and compared with 0.3 times the average relative standard deviation obtained from twenty-four elements which were evaluated in the previous two PTs Total Metals in Metal/Metal Alloy in agreement with the procedure of ISO13528, Annex B2 in the next table.

| | Cobalt as Co |
|---|---|
| RSD% observed | 5.1 |
| reference method | iis PTs |
| 0.3 x RSD% (reference method) | 6.6 |

Table 4: evaluation of the relative standard deviation of subsamples #23630-2

The calculated relative standard deviation is in agreement with 0.3 times the average relative standard deviation obtained from the evaluation of the elements from the previous two PTs. Therefore, homogeneity of #23630-2 was assumed.

To each of the participating laboratories one bracelet sample #23630 was sent on June 7, 2023.

### 2.5 ANALYZES

The participants were requested to determine on the parts of sample #23630: total levels of Antimony as Sb, Arsenic as As, Cadmium as Cd, Chromium as Cr, Cobalt as Co, Copper as Cu, Lead as Pb, Manganese as Mn, Mercury as Hg, Nickel as Ni, Selenium as Se, Strontium as Sr, Tin as Sn, Zinc as Zn and Zirconium as Zr.
It was requested to report if the laboratory was accredited for the determined elements and to report some analytical details.

It was explicitly requested to treat the sample as if it was a routine sample 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. Furthermore, some additional instructions were sent on how to measure the different parts of the bracelet sample and it was asked to report the test results for the different parts separately.

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 to 3 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 test result tables in appendices 1 to 3. 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 statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

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 requirements 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 and 2). 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.

This 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 appendices 1 to 2.

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 no problems were encountered with the dispatch of the samples. Six participants reported test results after the final reporting date and five other participants did not report any test results. Not all participants did not report all tests requested.
In total 48 participants reported 252 numerical test results. Observed were 14 outlying test results, which is 5.6%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

All data sets proved to have a normal Gaussian distribution.

### 4.1 EVALUATION PER PART OF THE SAMPLE AND PER ELEMENT

In this section the results are discussed per part of the 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 reported test results in appendices 1 and 2. The abbreviations, used in these tables, are explained in appendix 6.

Participants used different test methods to determine Metals in Metal/Metal Alloy. Some participants used test method CPSC-CH-E1001.08 (Determination of Total Lead in Metal Products), some used EN16711-1 (Determination of Metal content in Textile) or IEC62321-5 (Determination of Cadmium and Lead in Metals) and others used an in house method. Regretfully, no precision data is mentioned in CPSC-CH-E1001.08. The precision data mentioned in EN16711-1 involve a low concentration of metals tested in textile which may be not applicable here. The Horwitz equation can be used but it is a known fact that when Horwitz is used at higher concentrations, the equation returns a stricter reproducibility than at lower concentrations. Therefore, a target reproducibility based on the Horwitz equation may not be applicable to the higher concentrations of some metals in the bracelet.
However, method IEC62321-5 (published in 2014) does contain precision data based on a metal sample (metal parts found in e.g. buttons). The precision was determined for concentrations up to 1000 mg/kg and the RSD found for this range is 10%. It was decided to use this as the target reproducibility for all metals, calculated according to this formula:
0.1 * mean * 2.8.

**#23630-1: The decorated leaves at the ends of the bracelet**

Cadmium as Cd: This determination was not problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of IEC62321-5:14.

Copper as Cu: This determination was very problematic. No statistical outliers were observed. A large variation in the reported test results is observed, therefore no z-scores are calculated.

Lead as Pb: This determination was very problematic. In the leaves of the decorated ends of the bracelet solder is present. Solder usually consists of an alloy of Tin and Lead, although nowadays there are also lead-free variants. From the test results it seems that some participants included solder in the analysis while other participants did not. This resulted unfortunately in a large variation in the reported test results and therefore no z-scores are calculated, see also paragraph 5 for more discussion.

Tin as Sn: This determination was very problematic. Two statistical outliers were observed. In the leaves of the decorated ends of the bracelet solder is present. Solder usually consists of an alloy of Tin and Lead, although nowadays there are also lead-free variants. From the test results it seems that some participants included solder in the analysis while other participants did not. This resulted unfortunately in a large variation in the reported test results and therefore no z-scores are calculated.

Zinc as Zn: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of IEC62321-5:14.

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 3.

**#23630-2: Main part of the bracelet (wrist band)**

Arsenic as As: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of IEC62321-5:14.

Chromium as Cr: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of IEC62321-5:14.

Cobalt as Co: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of IEC62321-5:14.

Copper as Cu: This determination was very problematic. One statistical outlier was observed. A large variation in the reported test results is observed, therefore no z-scores are calculated.

Manganese as Mn: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of IEC62321-5:14.

Nickel as Ni: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of IEC62321-5:14.

Tin as Sn: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of IEC62321-5:14.

Zinc as Zn: This determination was very problematic. One statistical outlier was observed. A large variation in the reported test results is observed, therefore no z-scores are calculated.

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 3.

### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

| Element | unit | n | average | 2.8 * sd | R(lit) |
|---|---|---|---|---|---|
| Cadmium as Cd | mg/kg | 31 | 866561 | 127159 | 242637 |
| Copper as Cu | mg/kg | 15 | 53983 | 61391 | (15115) |
| Lead as Pb | mg/kg | 35 | n.e. | n.e. | n.e. |
| Tin as Sn | mg/kg | 11 | 500 | 436 | (140) |
| Zinc as Zn | mg/kg | 12 | 53775 | 12277 | (15057) |

Table 5: reproducibilities of tests on sample #23630-1 (decorated leaves, ends of the bracelet)

For results between brackets no z-scores are calculated.

| Element | unit | n | average | 2.8 * sd | R(lit) |
|---|---|---|---|---|---|
| Arsenic as As | mg/kg | 21 | 36.2 | 12.7 | 10.1 |
| Chromium as Cr | mg/kg | 30 | 216 | 89 | 60 |
| Cobalt as Co | mg/kg | 18 | 34.6 | 13.8 | 9.7 |
| Copper as Cu | mg/kg | 15 | 90327 | 109593 | (25292) |
| Manganese as Mn | mg/kg | 18 | 2782 | 732 | 779 |
| Nickel as Ni | mg/kg | 22 | 73.8 | 24.6 | 20.7 |
| Tin as Sn | mg/kg | 13 | 600 | 350 | 168 |
| Zinc as Zn | mg/kg | 12 | 167 | 338 | (47) |

Table 6: reproducibilities of tests on sample #23630-2 (main part of the bracelet (wrist band))

For results between brackets no z-scores are calculated.

Without further statistical calculations it can be concluded that for many tests there is not a good compliance of the group of participants with the reference test method. See also the discussion in paragraph 4.1 and 5.

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

| | June 2023 | April 2022 | June 2021 |
|---|---|---|---|
| Number of reporting laboratories | 48 | 66 | 56 |
| Number of test results | 252 | 627 | 350 |
| Number of statistical outliers | 14 | 59 | 26 |
| Percentage of statistical outliers | 5.6% | 9.4% | 7.4% |

Table 7: 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 | June 2023 | April 2022 | June 2021 |
|---|---|---|---|
| Arsenic as As | 12% | 20-42% | 20% |
| Cadmium as Cd | 5% | 17% | 15-17% |
| Chromium as Cr | 15% | 13-15% | 15-26% |
| Cobalt as Co | 14% | 12-17% | 20% |
| Copper as Cu | 41-43% | 4-25% | 10-42% |
| Lead as Pb | --- | 29% | 24-56% |
| Manganese as Mn | 9% | 6-8% | 5-9% |
| Nickel as Ni | 12% | 13-18% | 7-12% |
| Tin as Sn | 21-31% | 7-31% | --- |
| Zinc as Zn | 8-72% | 9-80% | 7-84% |

Table 8: development of the uncertainties over the years

The RSDs observed in this PT are in line with RSDs observed in previous iis PTs.

### 4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested which are listed in appendix 4. Based on the answers given by the participants the following can be summarized:

- 82% of the participants mentioned to be ISO/IEC17025 accredited to determine the reported elements for Metals in Metal/Metal Alloys.
- 88% of the participants used 0.5 grams or less as sample intake, most times 0.1 to 0.2 grams.
- 67% of the participants used Aqua Regia (a mixture of concentrated Nitric acid and concentrated Hydrochloric acid) as digestion acid, while 33% used Nitric acid only (in different concentrations).
- 60% of the participants used ICP-OES as technique to quantify the metals, 26% ICP-MS and 7% used AAS. A few used combinations of techniques with ICP-OES, ICP-MS and XRF.

As the majority of the group follow the same analytical procedures no further statistical analysis has been performed.

## 5 DISCUSSION

In this PT the average of the homogeneity test results for sample #23630-1 is not in line with the average (consensus value) from the PT results. There are several reasons for this. First, the goal of the homogeneity testing is very different from the goal of the evaluation of the reported PT results. To prove the homogeneity of the PT samples, a test method is selected with a high precision (smallest variation). The accuracy (trueness) of the test method is less relevant.
Secondly, the homogeneity testing is done by one laboratory only. The test results of this (ISO/IEC 17025 accredited) laboratory will have a bias (systematic deviation) depending on the test method used. The desire to detect small variations between the PT samples leads to the use of a sensitive test method with high precision, which may be a test method with significant bias. Also each test result reported by the laboratories that participate in the PT will have a bias. However, some will have a positive bias and others a negative bias. These different biases compensate each other in the PT average (consensus value). Therefore, the PT consensus value may deviate from the average of the homogeneity test. At the same time the accuracy of the PT consensus value is more reliable than the accuracy of the average of the results of the homogeneity test.

In this proficiency test for the determination of Metals in Metal/Metal Alloys a bracelet was purchased from a retailer and is therefore a real-life sample. Several participants contacted iis to ask how the leaves of the decoration of sample #23630-1 should be tested, specific whether the solder should be tested or not. The leaves were connected to the rest of the bracelet with apparently a Lead/Tin containing solder. In the letter of instructions iis requests participants to analyze the PT item in the way the laboratory is used to do. It appears that laboratories have different ways of working and some include (partly) the solder into the analysis while other laboratories skip the solder. As a result, the Lead and Tin determinations could not be evaluated because of the large deviation in reported test results. The large variation in these two elements may partly be explained by the lack of clear testing procedures on how to analyze this type of samples. It is recommended that members of technical committees bring this further into discussion to improve the analytical procedure in the test methods to avoid this confusion.

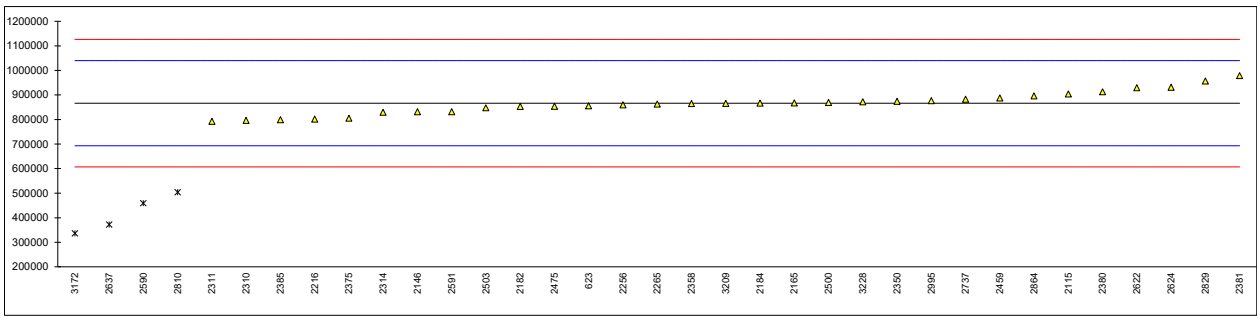
## 6 CONCLUSION

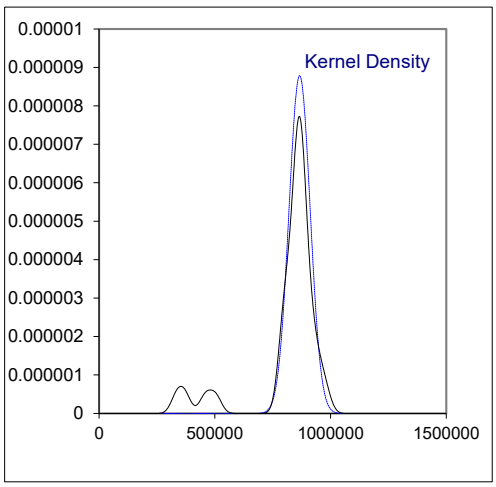
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

## APPENDIX 1 – Decoration leaves at the end of the bracelet

Determination of Cadmium as Cd on sample #23630-1; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 856126.27 | | -0.12 | |
| 840 | | ----- | | ----- | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 904146.2 | | 0.43 | |
| 2121 | | ----- | | ----- | |
| 2146 | In house | 831790 | | -0.40 | |
| 2159 | | ----- | | ----- | |
| 2165 | EN16711-1 | 867777 | | 0.01 | |
| 2182 | CPSC-CH-E1001-08.3 | 853061 | C | -0.16 | first reported 712078.3 |
| 2184 | CPSC-CH-E1001-08.3 | 867126 | | 0.01 | |
| 2216 | CPSC-CH-E1001-08.3 | 802135 | | -0.74 | |
| 2230 | | ----- | | ----- | |
| 2256 | CPSC-CH-E1001-08.3 | 860000 | | -0.08 | |
| 2265 | EN16711-1 | 863000 | | -0.04 | |
| 2310 | EN16711-1 | 796786 | | -0.81 | |
| 2311 | EN16711-1 | 792778.65 | | -0.85 | |
| 2314 | CPSC-CH-E1001-08.3 | 829521.18 | | -0.43 | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | CPSC-CH-E1001-08.3 | 874475 | | 0.09 | |
| 2358 | EN16711-1 | 864704 | | -0.02 | |
| 2366 | | ----- | | ----- | |
| 2373 | | ----- | | ----- | |
| 2375 | EN16711-1 | 805527 | | -0.70 | |
| 2380 | EN16711-1 | 912786.697 | | 0.53 | |
| 2381 | EN16711-1 | 979106.70 | | 1.30 | |
| 2382 | | ----- | | ----- | |
| 2385 | In house | 798753 | | -0.78 | |
| 2459 | EN16711-1 | 887800 | | 0.25 | |
| 2475 | In house | 854298.53 | | -0.14 | |
| 2500 | CPSC-CH-E1001-08.3 | 869799.5 | | 0.04 | |
| 2503 | | 848000 | C | -0.21 | first reported 848.000 |
| 2511 | | ----- | | ----- | |
| 2590 | CPSC-CH-E1001-08.3 | 459324.400 | C,R(0.01) | -4.70 | first reported 675442.28 |
| 2591 | CPSC-CH-E1001-08.3 | 831978.339 | | -0.40 | |
| 2622 | CPSC-CH-E1001-08.3 | 929220 | | 0.72 | |
| 2624 | In house | 932039 | | 0.76 | |
| 2637 | In house | 372000 | R(0.01) | -5.71 | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | CPSC-CH-E1001-08.3 | 882339.5 | | 0.18 | |
| 2741 | | ----- | | ----- | |
| 2810 | CPSC-CH-E1001-08.3 | 503849.39 | R(0.01) | -4.19 | |
| 2829 | CPSC-CH-E1001-08.3 | 957345.2 | | 1.05 | |
| 2864 | In house | 896380.85 | | 0.34 | |
| 2977 | | ----- | | ----- | |
| 2995 | EN62321-5 | 876342.95 | | 0.11 | |
| 3116 | | ----- | | ----- | |
| 3172 | IEC62321-3 | 336600 | R(0.01) | -6.12 | |
| 3209 | In house | 865965.51 | | -0.01 | |
| 3214 | | ----- | | ----- | |
| 3228 | CPSC-CH-E1001-08.3 | 872300 | | 0.07 | |
| | | | | | |
| | normality | OK | | | |
| | n | 31 | | | |
| | outliers | 4 | | | |
| | mean (n) | 866561.44 | | | |
| | st.dev. (n) | 45413.930 | RSD=5% | | |
| | R(calc.) | 127159.00 | | | |
| | st.dev.(IEC62321-5:14) | 86656.144 | | | |
| | R(IEC62321-5:14) | 242637.20 | | | |

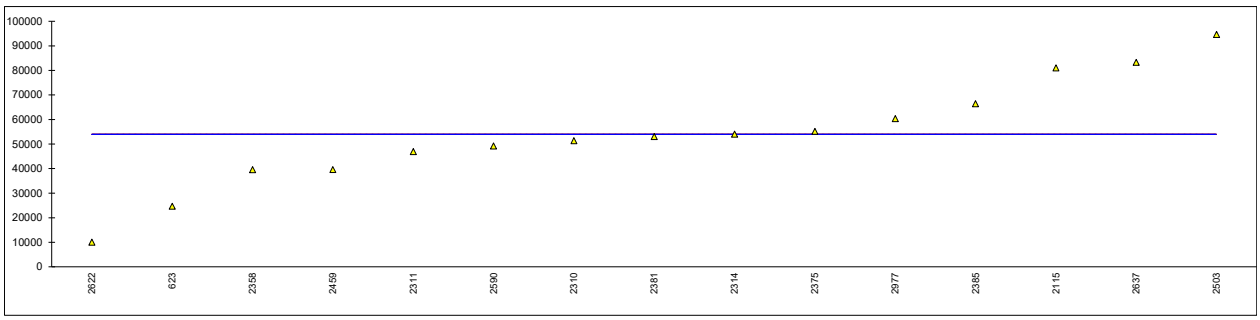

1200000
1100000
1000000
900000
800000
700000
600000
500000
400000
300000
200000
3172
2637
2590
2810
2311
2310
2385
2216
2375
2314
2146
2591
2503
2182
2475
623
2256
2265
2358
3209
2184
2165
2500
3228
2350
2995
2737
2459
2864
2115
2380
2622
2624
2829
2381

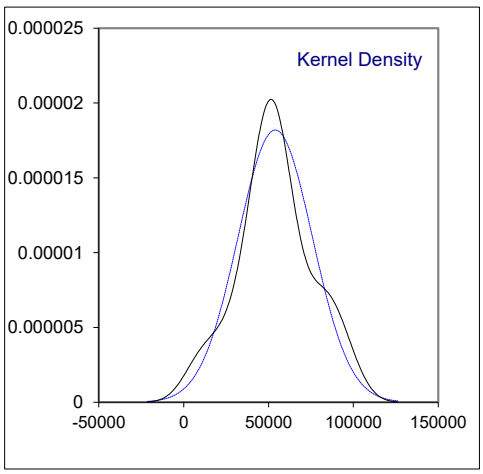

Kernel Density
0.00001
0.000009
0.000008
0.000007
0.000006
0.000005
0.000004
0.000003
0.000002
0.000001
0
0
500000
1000000
1500000

## Determination of Copper as Cu on sample #23630-1; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 24707.60 | | ----- | |
| 840 | | ----- | | ----- | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 81083 | C | ----- | first reported 181553.6 |
| 2121 | | ----- | | ----- | |
| 2146 | | ----- | | ----- | |
| 2159 | | ----- | | ----- | |
| 2165 | | ----- | | ----- | |
| 2182 | | ----- | | ----- | |
| 2184 | | ----- | | ----- | |
| 2216 | | ----- | | ----- | |
| 2230 | | ----- | | ----- | |
| 2256 | | ----- | | ----- | |
| 2265 | | ----- | | ----- | |
| 2310 | EN16711-1 | 51367 | | ----- | |
| 2311 | EN16711-1 | 46989.74 | | ----- | |
| 2314 | CPSC-CH-E1001-08.3 | 54094.40 | | ----- | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | | ----- | | ----- | |
| 2358 | EN16711-1 | 39560 | | ----- | |
| 2366 | | ----- | | ----- | |
| 2373 | | ----- | | ----- | |
| 2375 | EN16711-1 | 55210 | | ----- | |
| 2380 | | ----- | | ----- | |
| 2381 | EN16711-1 | 53085.1 | C | ----- | first reported 126109.60 |
| 2382 | | ----- | | ----- | |
| 2385 | In house | 66398 | | ----- | |
| 2459 | EN16711-1 | 39625 | | ----- | |
| 2475 | | ----- | | ----- | |
| 2500 | | ----- | | ----- | |
| 2503 | | 94700 | C | ----- | first reported 94.700 |
| 2511 | | ----- | | ----- | |
| 2590 | CPSC-CH-E1001-08.3 | 49251.83 | | ----- | |
| 2591 | | ----- | | ----- | |
| 2622 | CPSC-CH-E1001-08.3 | 10029 | | ----- | |
| 2624 | | ----- | | ----- | |
| 2637 | In house | 83300 | | ----- | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | | ----- | | ----- | |
| 2741 | | ----- | | ----- | |
| 2810 | | ----- | | ----- | |
| 2829 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2977 | CPSC-CH-E1001-08.3 | 60349 | | ----- | |
| 2995 | | ----- | | ----- | |
| 3116 | | ----- | | ----- | |
| 3172 | | ----- | | ----- | |
| 3209 | | ----- | | ----- | |
| 3214 | | ----- | | ----- | |
| 3228 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 15 |
| outliers | 0 |
| mean (n) | 53983.31 |
| st.dev. (n) | 21925.303 |
| R(calc.) | 61390.85 |
| st.dev.(IEC62321-5:14) | (5398.331) |
| R(IEC62321-5:14) | (15115.33) |


100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
2622
623
2358
2459
2311
2590
2310
2381
2314
2375
2977
2385
2115
2637
2503

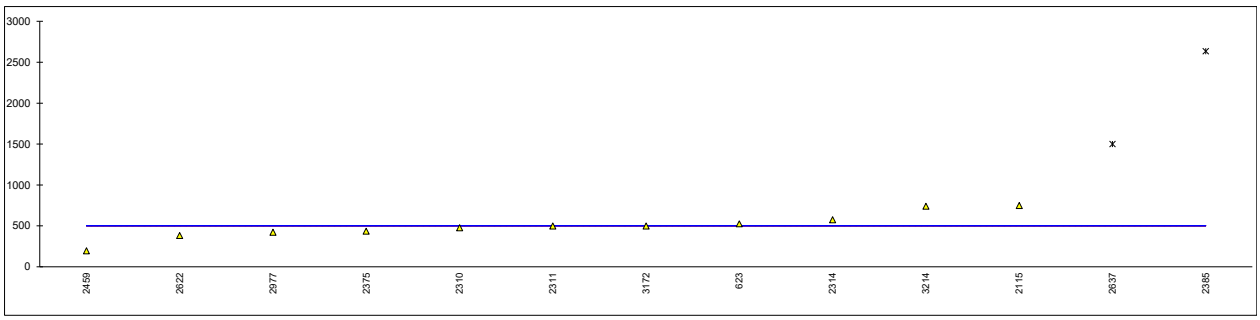

Kernel Density
0.000025
0.00002
0.000015
0.00001
0.000005
0
-50000
0
50000
100000
150000

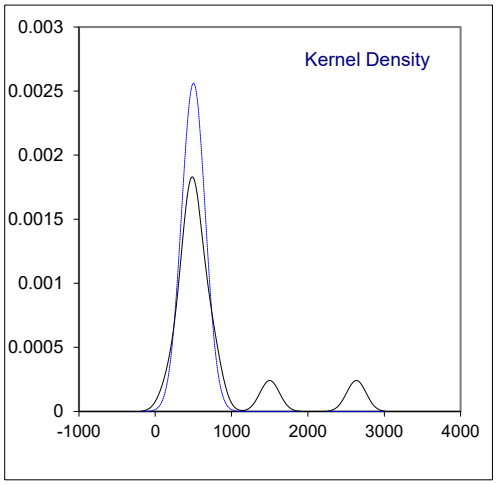
Determination of Lead as Pb on sample #23630-1; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 172.70 | | ----- | |
| 840 | | ----- | | ----- | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 6.18 | | ----- | |
| 2121 | CPSC-CH-E1001-08.3 | 6.554 | | ----- | |
| 2146 | In house | 22537 | | ----- | |
| 2159 | | ----- | | ----- | |
| 2165 | EN16711-1 | Not detected | | ----- | |
| 2182 | CPSC-CH-E1001-08.3 | not detected | | ----- | |
| 2184 | CPSC-CH-E1001-08.3 | <10 | | ----- | |
| 2216 | CPSC-CH-E1001-08.3 | 26018 | | ----- | |
| 2230 | CPSC-CH-E1001-08.3 | <10 | | ----- | |
| 2256 | CPSC-CH-E1001-08.3 | Not detected | | ----- | |
| 2265 | EN16711-1 | 3940 | | ----- | |
| 2310 | EN16711-1 | <10 | | ----- | |
| 2311 | EN16711-1 | 11.97 | | ----- | |
| 2314 | | ----- | | ----- | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | CPSC-CH-E1001-08.3 | < 50 | | ----- | |
| 2358 | EN16711-1 | not detected | | ----- | |
| 2366 | CPSC-CH-E1001-08.3 | 15309 | | ----- | |
| 2373 | | ----- | | ----- | |
| 2375 | EN16711-1 | 6400 | | ----- | |
| 2380 | EN16711-1 | 13.196 | | ----- | |
| 2381 | EN16711-1 | 14.90 | | ----- | |
| 2382 | CPSC-CH-E1001-08.3 | 6600 | | ----- | |
| 2385 | In house | 11187 | | ----- | |
| 2459 | CPSC-CH-E1001-08.3 | 346.53 | | ----- | |
| 2475 | | ----- | | ----- | |
| 2500 | CPSC-CH-E1001-08.3 | ND | | ----- | |
| 2503 | | ----- | | ----- | |
| 2511 | CPSC-CH-E1001-08.3 | <10 | | ----- | |
| 2590 | CPSC-CH-E1001-08.3 | 3.48 | | ----- | |
| 2591 | CPSC-CH-E1001-08.3 | 56984.162 | | ----- | |
| 2622 | | ----- | | ----- | |
| 2624 | | ----- | | ----- | |
| 2637 | In house | 6320 | | ----- | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | | ----- | | ----- | |
| 2741 | | ----- | | ----- | |
| 2810 | CPSC-CH-E1001-08.3 | 14374.76 | | ----- | |
| 2829 | CPSC-CH-E1001-08.3 | not detected | | ----- | |
| 2864 | In house | 8.35 | | ----- | |
| 2977 | CPSC-CH-E1001-08.3 | not detected | | ----- | |
| 2995 | EN62321-5 | 8731.83 | | ----- | |
| 3116 | CPSC-CH-E1001-08.3 | <10 | | ----- | |
| 3172 | | ----- | | ----- | |
| 3209 | In house | <10.0 | | ----- | |
| 3214 | EN16711-1 | <10 | | ----- | |
| 3228 | CPSC-CH-E1001-08.3 | <10 | | ----- | |
| | n | 24 | 11 | | |
| | mean (n) | <350 | >3500 | | |

## Determination of Tin as Sn on sample #23630-1; results in mg/kg

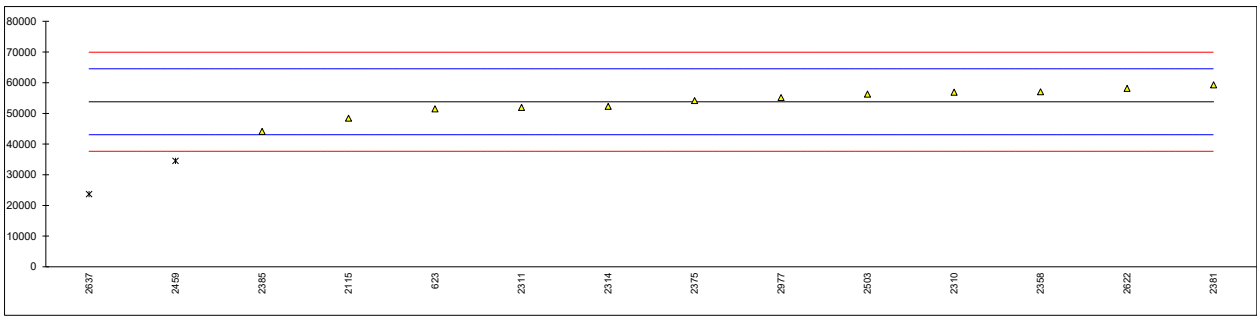
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 525.29 | | ----- | |
| 840 | | ----- | | ----- | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 749.3 | | ----- | |
| 2121 | | ----- | | ----- | |
| 2146 | | ----- | | ----- | |
| 2159 | | ----- | | ----- | |
| 2165 | | ----- | | ----- | |
| 2182 | | ----- | | ----- | |
| 2184 | | ----- | | ----- | |
| 2216 | | ----- | | ----- | |
| 2230 | | ----- | | ----- | |
| 2256 | | ----- | | ----- | |
| 2265 | | ----- | | ----- | |
| 2310 | EN16711-1 | 478 | | ----- | |
| 2311 | EN16711-1 | 498.43 | | ----- | |
| 2314 | CPSC-CH-E1001-08.3 | 574.21 | | ----- | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | | ----- | | ----- | |
| 2358 | EN16711-1 | not detected | C | ----- | first reported 167 / possibly a false negative test result? |
| 2366 | | ----- | | ----- | |
| 2373 | | ----- | | ----- | |
| 2375 | EN16711-1 | 435 | | ----- | |
| 2380 | | ----- | | ----- | |
| 2381 | | ----- | | ----- | |
| 2382 | | ----- | | ----- | |
| 2385 | In house | 2634 | G(0.01) | ----- | |
| 2459 | EN16711-1 | 196.36 | | ----- | |
| 2475 | | ----- | | ----- | |
| 2500 | | ----- | | ----- | |
| 2503 | | ----- | | ----- | |
| 2511 | | ----- | | ----- | |
| 2590 | | ----- | | ----- | |
| 2591 | | ----- | | ----- | |
| 2622 | CPSC-CH-E1001-08.3 | 382 | | ----- | |
| 2624 | | ----- | | ----- | |
| 2637 | In house | 1500 | G(0.01) | ----- | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | | ----- | | ----- | |
| 2741 | | ----- | | ----- | |
| 2810 | | ----- | | ----- | |
| 2829 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2977 | CPSC-CH-E1001-08.3 | 422.9 | | ----- | |
| 2995 | | ----- | | ----- | |
| 3116 | | ----- | | ----- | |
| 3172 | EN16711-1 | 499 | | ----- | |
| 3209 | | ----- | | ----- | |
| 3214 | EN16711-1 | 740.34 | | ----- | |
| 3228 | | ----- | | ----- | |
| | normality | OK | | | |
| | n | 11 | | | |
| | outliers | 2 | | | |
| | mean (n) | 500.075 | | | |
| | st.dev. (n) | 155.8328 | RSD=31% | | |
| | R(calc.) | 436.332 | | | |
| | st.dev.(IEC62321-5:14) | (50.0075) | | | |
| | R(IEC62321-5:14) | (140.021) | | | |

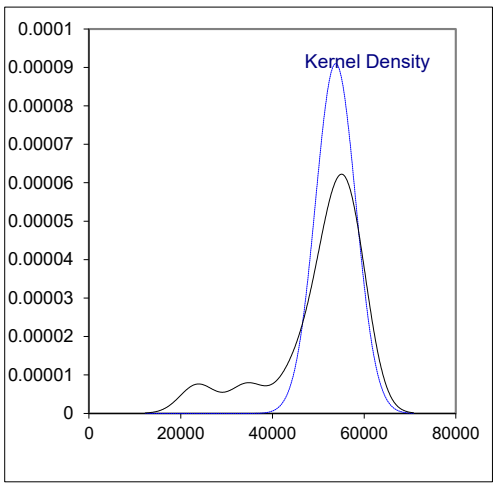

3000
2500
2000
1500
1000
500
0
2459
2622
2977
2375
2310
2311
3172
623
2314
3214
2115
2637
2385


Kernel Density
0.003
0.0025
0.002
0.0015
0.001
0.0005
0
-1000
0
1000
2000
3000
4000

Determination of Zinc as Zn on sample #23630-1; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 51490.35 | | -0.42 | |
| 840 | | ----- | | ----- | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 48431.6 | | -0.99 | |
| 2121 | | ----- | | ----- | |
| 2146 | | ----- | | ----- | |
| 2159 | | ----- | | ----- | |
| 2165 | | ----- | | ----- | |
| 2182 | | ----- | | ----- | |
| 2184 | | ----- | | ----- | |
| 2216 | | ----- | | ----- | |
| 2230 | | ----- | | ----- | |
| 2256 | | ----- | | ----- | |
| 2265 | | ----- | | ----- | |
| 2310 | EN16711-1 | 56921 | | 0.59 | |
| 2311 | EN16711-1 | 51959.61 | | -0.34 | |
| 2314 | CPSC-CH-E1001-08.3 | 52302.14 | | -0.27 | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | | ----- | | ----- | |
| 2358 | EN16711-1 | 57044 | | 0.61 | |
| 2366 | | ----- | | ----- | |
| 2373 | | ----- | | ----- | |
| 2375 | EN16711-1 | 54166 | | 0.07 | |
| 2380 | | ----- | | ----- | |
| 2381 | EN16711-1 | 59275.50 | | 1.02 | |
| 2382 | | ----- | | ----- | |
| 2385 | In house | 44091 | | -1.80 | |
| 2459 | EN16711-1 | 34502.22 | G(0.05) | -3.58 | |
| 2475 | | ----- | | ----- | |
| 2500 | | ----- | | ----- | |
| 2503 | | 56300 | C | 0.47 | first reported 56.300 |
| 2511 | | ----- | | ----- | |
| 2590 | | ----- | | ----- | |
| 2591 | | ----- | | ----- | |
| 2622 | CPSC-CH-E1001-08.3 | 58151 | | 0.81 | |
| 2624 | | ----- | | ----- | |
| 2637 | In house | 23700 | G(0.05) | -5.59 | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | | ----- | | ----- | |
| 2741 | | ----- | | ----- | |
| 2810 | | ----- | | ----- | |
| 2829 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2977 | CPSC-CH-E1001-08.3 | 55167 | | 0.26 | |
| 2995 | | ----- | | ----- | |
| 3116 | | ----- | | ----- | |
| 3172 | | ----- | | ----- | |
| 3209 | | ----- | | ----- | |
| 3214 | | ----- | | ----- | |
| 3228 | | ----- | | ----- | |
| | normality | OK | | | |
| | n | 12 | | | |
| | outliers | 2 | | | |
| | mean (n) | 53774.93 | | | |
| | st.dev. (n) | 4384.591 | RSD=8% | | |
| | R(calc.) | 12276.86 | | | |
| | st.dev.(IEC62321-5:14) | 5377.493 | | | |
| | R(IEC62321-5:14) | 15056.98 | | | |


80000
70000
60000
50000
40000
30000
20000
10000
0
2637
2459
2385
2115
623
2311
2314
2375
2977
2503
2310
2358
2622
2381


Kernel Density
0.0001
0.00009
0.00008
0.00007
0.00006
0.00005
0.00004
0.00003
0.00002
0.00001
0
0
20000
40000
60000
80000

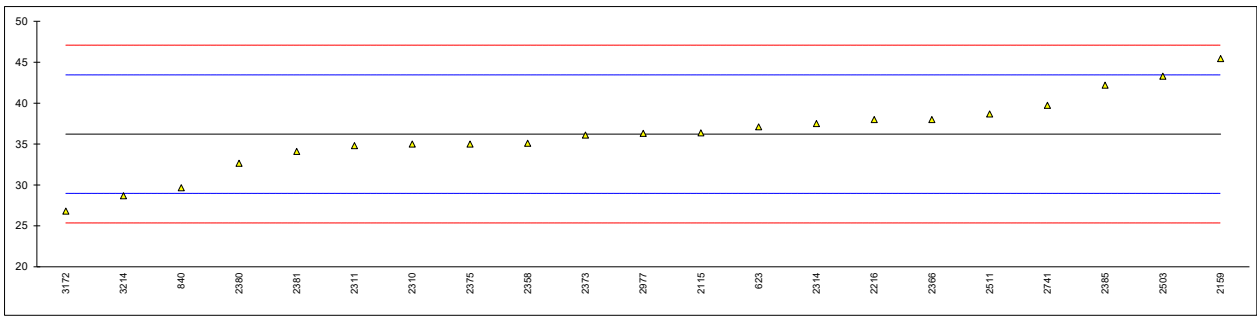
## APPENDIX 2 – Main part of the bracelet

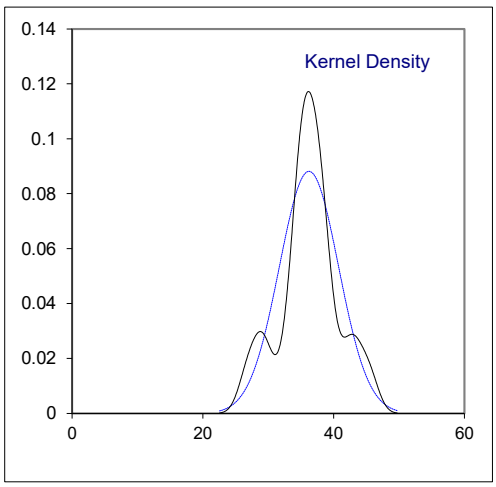
Determination of Arsenic as As on sample #23630-2; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 37.10 | | 0.24 | |
| 840 | In house | 29.66 | | -1.81 | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 36.36 | | 0.04 | |
| 2121 | | ----- | | ----- | |
| 2146 | | ----- | | ----- | |
| 2159 | In house | 45.44 | | 2.55 | |
| 2165 | | ----- | | ----- | |
| 2182 | | ----- | | ----- | |
| 2184 | | ----- | | ----- | |
| 2216 | CPSC-CH-E1001-08.3 | 38 | | 0.49 | |
| 2230 | | ----- | | ----- | |
| 2256 | | ----- | | ----- | |
| 2265 | EN16711-1 | not detected | | ----- | possibly a false negative test result? |
| 2310 | EN16711-1 | 35 | | -0.34 | |
| 2311 | EN16711-1 | 34.81 | | -0.39 | |
| 2314 | EN16711-1 | 37.51 | | 0.36 | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | | ----- | | ----- | |
| 2358 | EN16711-1 | 35.1 | | -0.31 | |
| 2366 | CPSC-CH-E1001-08.3 | 38 | | 0.49 | |
| 2373 | EN16711-1 | 36.1 | | -0.03 | |
| 2375 | EN16711-1 | 35 | | -0.34 | |
| 2380 | EN16711-1 | 32.669 | | -0.98 | |
| 2381 | EN16711-1 | 34.10 | | -0.58 | |
| 2382 | | ----- | | ----- | |
| 2385 | In house | 42.2 | | 1.65 | |
| 2459 | | ----- | | ----- | |
| 2475 | | ----- | | ----- | |
| 2500 | | ----- | | ----- | |
| 2503 | CPSC-CH-E1001-08.3 | 43.3 | | 1.96 | |
| 2511 | EN16711-1 | 38.681 | | 0.68 | |
| 2590 | CPSC-CH-E1001-08.3 | < L.O.Q. | | ----- | |
| 2591 | | ----- | | ----- | |
| 2622 | | ----- | | ----- | |
| 2624 | | ----- | | ----- | |
| 2637 | | ----- | | ----- | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | | ----- | | ----- | |
| 2741 | CPSC-CH-E1001-08.3 | 39.733 | | 0.97 | |
| 2810 | | ----- | | ----- | |
| 2829 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2977 | CPSC-CH-E1001-08.3 | 36.32 | | 0.03 | |
| 2995 | | ----- | | ----- | |
| 3116 | | ----- | | ----- | |
| 3172 | EN16711-1 | 26.8 | | -2.60 | |
| 3209 | | ----- | | ----- | |
| 3214 | EN16711-1 | 28.70 | | -2.08 | |
| 3228 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 21 |
| outliers | 0 |
| mean (n) | 36.2182 |
| st.dev. (n) | 4.52634 |
| R(calc.) | 12.6738 |
| st.dev.(IEC62321-5:14) | 3.62182 |
| R(IEC62321-5:14) | 10.1411 |

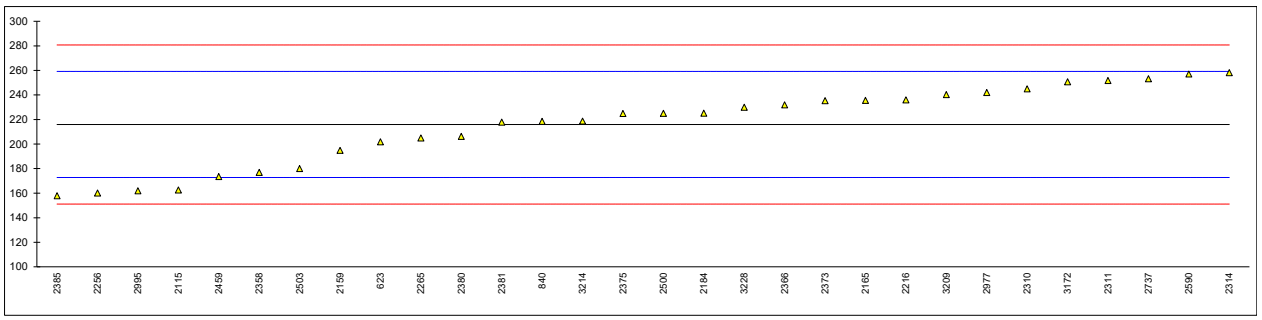
RSD=12%

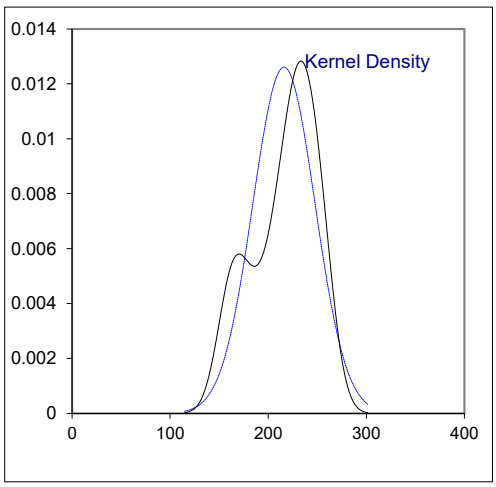

50
45
40
35
30
25
20
3172
3214
840
2380
2381
2311
2310
2375
2358
2373
2977
2115
623
2314
2216
2366
2511
2741
2385
2503
2159


Kernel Density
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
0
20
40
60

Determination of Chromium as Cr on sample #23630-2; results in mg/kg

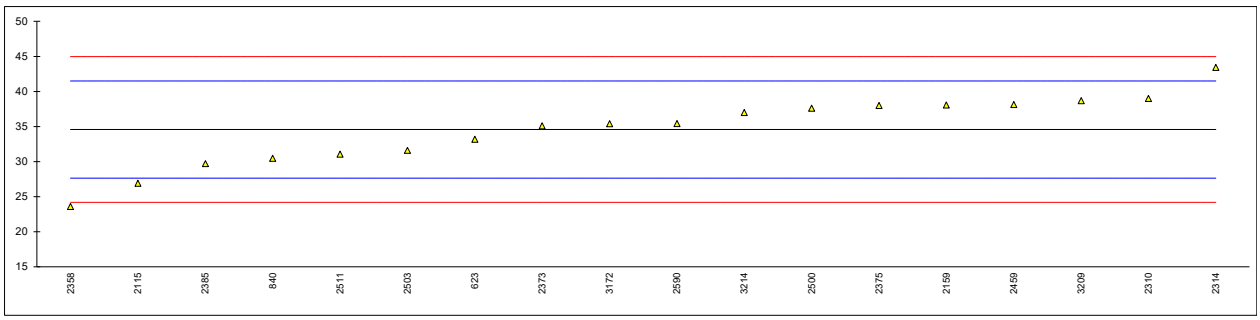
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 201.85 | | -0.65 | |
| 840 | In house | 218.51 | | 0.12 | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 162.63 | | -2.47 | |
| 2121 | | ----- | | ----- | |
| 2146 | | ----- | | ----- | |
| 2159 | In house | 194.9 | | -0.98 | |
| 2165 | EN16711-1 | 235.7 | | 0.91 | |
| 2182 | | ----- | | ----- | |
| 2184 | CPSC-CH-E1001-08.3 | 225.2 | | 0.43 | |
| 2216 | CPSC-CH-E1001-08.3 | 236 | | 0.93 | |
| 2230 | | ----- | | ----- | |
| 2256 | CPSC-CH-E1001-08.3 | 160.025 | | -2.59 | |
| 2265 | EN16711-1 | 205.0 | | -0.51 | |
| 2310 | EN16711-1 | 245 | | 1.34 | |
| 2311 | EN16711-1 | 251.78 | | 1.66 | |
| 2314 | EN16711-1 | 258.14 | | 1.95 | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | | ----- | | ----- | |
| 2358 | EN16711-1 | 177 | | -1.80 | |
| 2366 | CPSC-CH-E1001-08.3 | 232 | | 0.74 | |
| 2373 | EN16711-1 | 235.3 | | 0.90 | |
| 2375 | EN16711-1 | 225 | | 0.42 | |
| 2380 | EN16711-1 | 206.274 | | -0.45 | |
| 2381 | EN16711-1 | 217.90 | | 0.09 | |
| 2382 | | ----- | | ----- | |
| 2385 | In house | 158 | | -2.68 | |
| 2459 | EN16711-1 | 173.6 | | -1.96 | |
| 2475 | | ----- | | ----- | |
| 2500 | CPSC-CH-E1001-08.3 | 225.1 | | 0.42 | |
| 2503 | CPSC-CH-E1001-08.3 | 180 | | -1.67 | |
| 2511 | | ----- | | ----- | |
| 2590 | CPSC-CH-E1001-08.3 | 257.071 | C | 1.90 | first reported 49.58 |
| 2591 | | ----- | | ----- | |
| 2622 | | ----- | | ----- | |
| 2624 | | ----- | | ----- | |
| 2637 | | ----- | | ----- | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | CPSC-CH-E1001-08.3 | 253.19 | | 1.72 | |
| 2741 | | ----- | | ----- | |
| 2810 | | ----- | | ----- | |
| 2829 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2977 | CPSC-CH-E1001-08.3 | 242.0 | | 1.21 | |
| 2995 | EN62321-5 | 162 | | -2.50 | |
| 3116 | | ----- | | ----- | |
| 3172 | EN16711-1 | 250.7 | | 1.61 | |
| 3209 | In house | 240.36 | | 1.13 | |
| 3214 | EN16711-1 | 218.75 | | 0.13 | |
| 3228 | CPSC-CH-E1001-08.3 | 230 | | 0.65 | |
| | normality | OK | | | |
| | n | 30 | | | |
| | outliers | 0 | | | |
| | mean (n) | 215.9660 | | | |
| | st.dev. (n) | 31.65342 | RSD=15% | | |
| | R(calc.) | 88.6296 | | | |
| | st.dev.(IEC62321-5:14) | 21.59660 | | | |
| | R(IEC62321-5:14) | 60.4705 | | | |

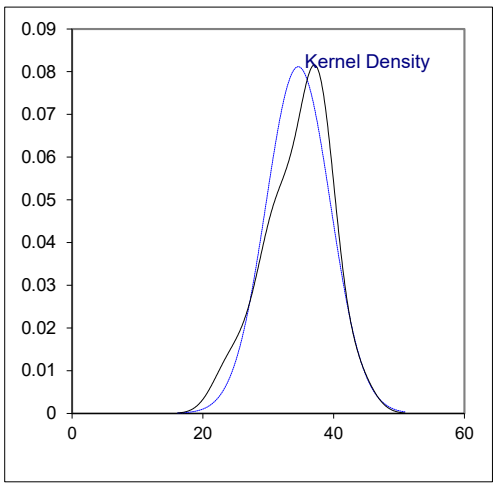

300
280
260
240
220
200
180
160
140
120
100
2385
2256
2995
2115
2459
2358
2503
2159
623
2265
2360
2381
840
3214
2375
2500
2184
3228
2366
2373
2165
2216
3209
2977
2310
3172
2311
2737
2590
2314


Kernel Density
0.014
0.012
0.01
0.008
0.006
0.004
0.002
0
0
100
200
300
400

## Determination of Cobalt as Co on sample #23630-2; results in mg/kg

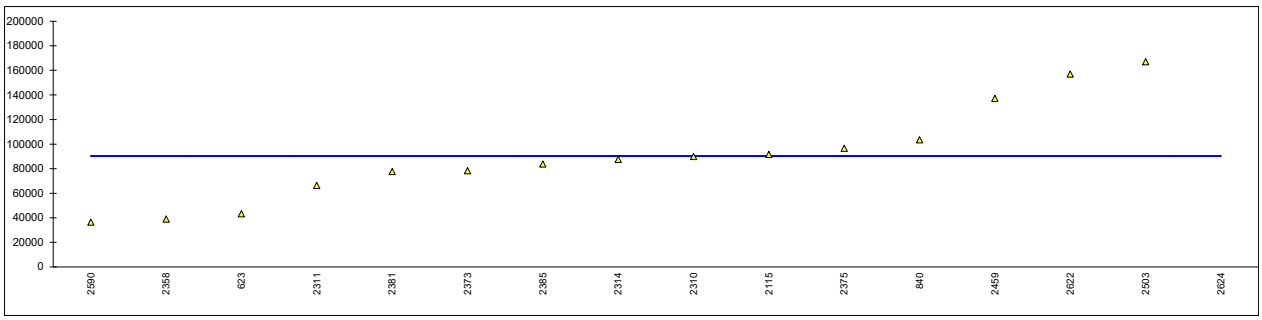
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 33.19 | | -0.40 | |
| 840 | In house | 30.46 | | -1.19 | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 26.92 | | -2.21 | |
| 2121 | | ----- | | ----- | |
| 2146 | | ----- | | ----- | |
| 2159 | In house | 38.068 | | 1.01 | |
| 2165 | | ----- | | ----- | |
| 2182 | | ----- | | ----- | |
| 2184 | | ----- | | ----- | |
| 2216 | | ----- | | ----- | |
| 2230 | | ----- | | ----- | |
| 2256 | | ----- | | ----- | |
| 2265 | | ----- | | ----- | |
| 2310 | EN16711-1 | 39 | | 1.28 | |
| 2311 | EN16711-1 | Not Detected | | ----- | possibly a false negative test result? |
| 2314 | EN16711-1 | 43.43 | | 2.56 | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | | ----- | | ----- | |
| 2358 | EN16711-1 | 23.6 | | -3.17 | |
| 2366 | | ----- | | ----- | |
| 2373 | EN16711-1 | 35.1 | | 0.15 | |
| 2375 | EN16711-1 | 38 | | 0.99 | |
| 2380 | | ----- | | ----- | |
| 2381 | | ----- | | ----- | |
| 2382 | | ----- | | ----- | |
| 2385 | In house | 29.7 | | -1.41 | |
| 2459 | EN16711-1 | 38.14 | | 1.03 | |
| 2475 | | ----- | | ----- | |
| 2500 | CPSC-CH-E1001-08.3 | 37.6 | | 0.87 | |
| 2503 | CPSC-CH-E1001-08.3 | 31.6 | | -0.86 | |
| 2511 | EN16711-1 | 31.076 | | -1.01 | |
| 2590 | CPSC-CH-E1001-08.3 | 35.42 | | 0.24 | |
| 2591 | | ----- | | ----- | |
| 2622 | | ----- | | ----- | |
| 2624 | | ----- | | ----- | |
| 2637 | | ----- | | ----- | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | | ----- | | ----- | |
| 2741 | | ----- | | ----- | |
| 2810 | | ----- | | ----- | |
| 2829 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2977 | CPSC-CH-E1001-08.3 | not detected | | ----- | possibly a false negative test result? |
| 2995 | | ----- | | ----- | |
| 3116 | | ----- | | ----- | |
| 3172 | EN16711-1 | 35.4 | | 0.24 | |
| 3209 | In house | 38.68 | | 1.19 | |
| 3214 | EN16711-1 | 36.99 | | 0.70 | |
| 3228 | | ----- | | ----- | |
| | normality | OK | | | |
| | n | 18 | | | |
| | outliers | 0 | | | |
| | mean (n) | 34.5763 | | | |
| | st.dev. (n) | 4.91634 | RSD=14% | | |
| | R(calc.) | 13.7658 | | | |
| | st.dev.(IEC62321-5:14) | 3.45763 | | | |
| | R(IEC62321-5:14) | 9.6814 | | | |

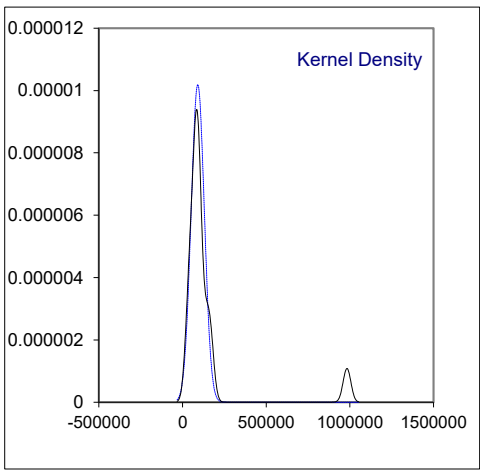

50
45
40
35
30
25
20
15
2388
2115
2385
840
2511
2503
623
2373
3172
2590
3214
2500
2375
2159
2459
3209
2310
2314


Kernel Density
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
0
20
40
60

## Determination of Copper as Cu on sample #23630-2; results in mg/kg

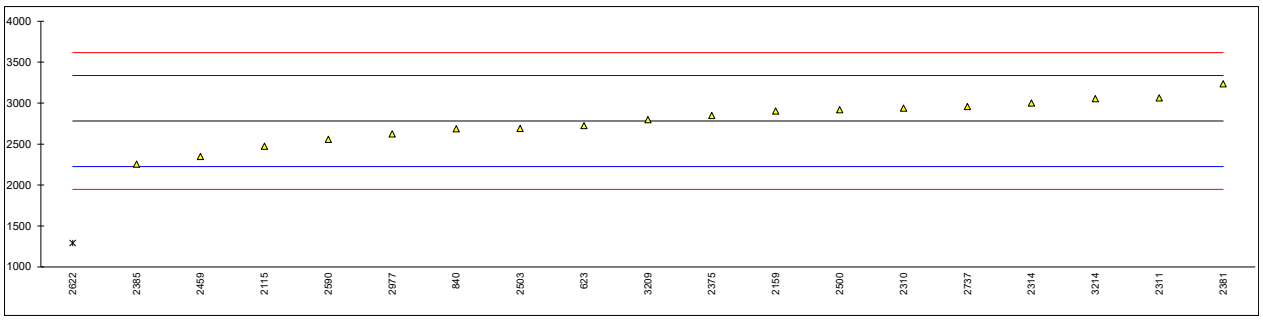
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 43233.86 | | ----- | |
| 840 | In house | 103506.6 | | ----- | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 91669 | C | ----- | first reported 208622.2 |
| 2121 | | ----- | | ----- | |
| 2146 | | ----- | | ----- | |
| 2159 | | ----- | | ----- | |
| 2165 | | ----- | | ----- | |
| 2182 | | ----- | | ----- | |
| 2184 | | ----- | | ----- | |
| 2216 | | ----- | | ----- | |
| 2230 | | ----- | | ----- | |
| 2256 | | ----- | | ----- | |
| 2265 | | ----- | | ----- | |
| 2310 | EN16711-1 | 89825 | | ----- | |
| 2311 | EN16711-1 | 66229.66 | | ----- | |
| 2314 | EN16711-1 | 87532.43 | | ----- | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | | ----- | | ----- | |
| 2358 | EN16711-1 | 38916 | | ----- | |
| 2366 | | ----- | | ----- | |
| 2373 | EN16711-1 | 78346.3 | | ----- | |
| 2375 | EN16711-1 | 96609 | | ----- | |
| 2380 | | ----- | | ----- | |
| 2381 | EN16711-1 | 77582.50 | | ----- | |
| 2382 | | ----- | | ----- | |
| 2385 | In house | 83810 | | ----- | |
| 2459 | EN16711-1 | 137253.32 | | ----- | |
| 2475 | | ----- | | ----- | |
| 2500 | | ----- | | ----- | |
| 2503 | CPSC-CH-E1001-08.3 | 167000 | C | ----- | first reported 167.000 |
| 2511 | | ----- | | ----- | |
| 2590 | CPSC-CH-E1001-08.3 | 36396.41 | | ----- | |
| 2591 | | ----- | | ----- | |
| 2622 | | 157000 | | ----- | |
| 2624 | In house | 981930 | G(0.01) | ----- | |
| 2637 | | ----- | | ----- | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | | ----- | | ----- | |
| 2741 | | ----- | | ----- | |
| 2810 | | ----- | | ----- | |
| 2829 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2977 | CPSC-CH-E1001-08.3 | not detected | | ----- | possibly a false negative test result? |
| 2995 | | ----- | | ----- | |
| 3116 | | ----- | | ----- | |
| 3172 | | ----- | | ----- | |
| 3209 | | ----- | | ----- | |
| 3214 | | ----- | | ----- | |
| 3228 | | ----- | | ----- | |
| | normality | OK | | | |
| | n | 15 | | | |
| | outliers | 1 | | | |
| | mean (n) | 90327.34 | | | |
| | st.dev. (n) | 39140.426 | RSD=43% | | |
| | R(calc.) | 109593.19 | | | |
| | st.dev.(IEC62321-5:14) | (9032.734) | | | |
| | R(IEC62321-5:14) | (25291.66) | | | |

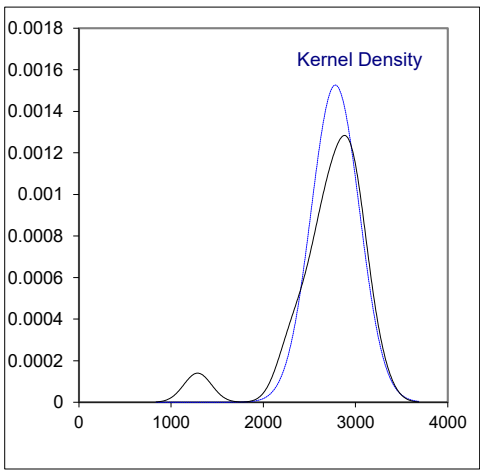

200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
2590
2358
623
2311
2381
2373
2385
2314
2310
2115
2375
840
2459
2622
2503
2624


Kernel Density
0.000012
0.00001
0.000008
0.000006
0.000004
0.000002
0
-500000
0
500000
1000000
1500000

Determination of Manganese as Mn on sample #23630-2; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 2724.64 | | -0.21 | |
| 840 | In house | 2685.61 | | -0.35 | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 2474.4 | | -1.10 | |
| 2121 | | ----- | | ----- | |
| 2146 | | ----- | | ----- | |
| 2159 | In house | 2903 | | 0.44 | |
| 2165 | | ----- | | ----- | |
| 2182 | | ----- | | ----- | |
| 2184 | | ----- | | ----- | |
| 2216 | | ----- | | ----- | |
| 2230 | | ----- | | ----- | |
| 2256 | | ----- | | ----- | |
| 2265 | | ----- | | ----- | |
| 2310 | EN16711-1 | 2938 | | 0.56 | |
| 2311 | EN16711-1 | 3062.84 | | 1.01 | |
| 2314 | EN16711-1 | 3002.25 | | 0.79 | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | | ----- | | ----- | |
| 2358 | | ----- | | ----- | |
| 2366 | | ----- | | ----- | |
| 2373 | | ----- | | ----- | |
| 2375 | EN16711-1 | 2847 | | 0.23 | |
| 2380 | | ----- | | ----- | |
| 2381 | EN16711-1 | 3235.60 | | 1.63 | |
| 2382 | | ----- | | ----- | |
| 2385 | In house | 2254 | | -1.90 | |
| 2459 | EN16711-1 | 2346.25 | | -1.57 | |
| 2475 | | ----- | | ----- | |
| 2500 | CPSC-CH-E1001-08.3 | 2919.1 | | 0.49 | |
| 2503 | CPSC-CH-E1001-08.3 | 2690 | C | -0.33 | first reported 2.690 |
| 2511 | | ----- | | ----- | |
| 2590 | CPSC-CH-E1001-08.3 | 2556.55 | | -0.81 | |
| 2591 | | ----- | | ----- | |
| 2622 | | 1289 | G(0.01) | -5.37 | |
| 2624 | | ----- | | ----- | |
| 2637 | | ----- | | ----- | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | CPSC-CH-E1001-08.3 | 2958.47 | | 0.64 | |
| 2741 | | ----- | | ----- | |
| 2810 | | ----- | | ----- | |
| 2829 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2977 | CPSC-CH-E1001-08.3 | 2622 | | -0.57 | |
| 2995 | | ----- | | ----- | |
| 3116 | | ----- | | ----- | |
| 3172 | | ----- | | ----- | |
| 3209 | In house | 2797.01 | | 0.05 | |
| 3214 | EN16711-1 | 3054.85 | | 0.98 | |
| 3228 | | ----- | | ----- | |
| | normality | OK | | | |
| | n | 18 | | | |
| | outliers | 1 | | | |
| | mean (n) | 2781.754 | | | |
| | st.dev. (n) | 261.4100 | RSD=9% | | |
| | R(calc.) | 731.948 | | | |
| | st.dev.(IEC62321-5:14) | 278.1754 | | | |
| | R(IEC62321-5:14) | 778.891 | | | |

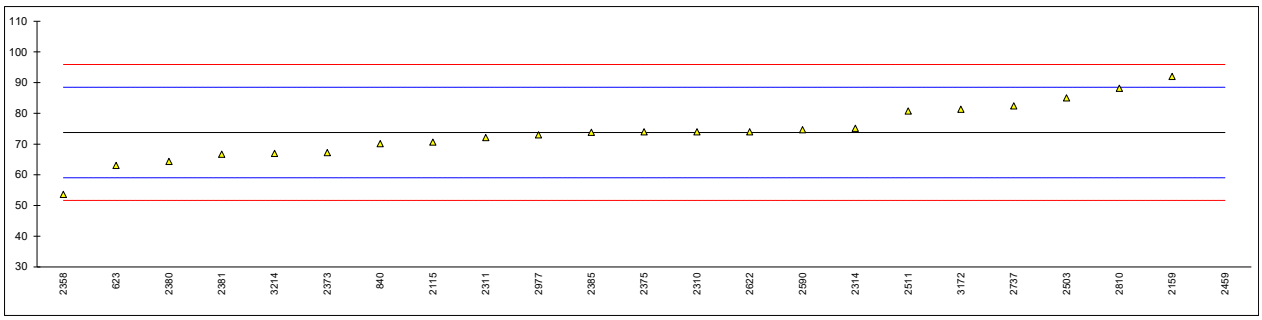

4000
3500
3000
2500
2000
1500
1000
2622
2385
2459
2115
2590
2977
840
2503
623
3209
2375
2159
2500
2310
2737
2314
3214
2311
2381

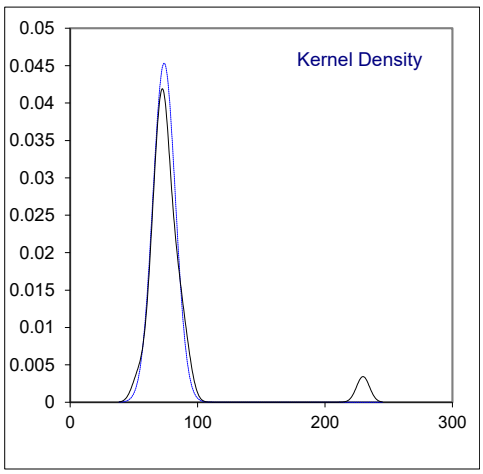

Kernel Density
0.0018
0.0016
0.0014
0.0012
0.001
0.0008
0.0006
0.0004
0.0002
0
0
1000
2000
3000
4000

## Determination of Nickel as Ni on sample #23630-2; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 63.03 | | -1.46 | |
| 840 | In house | 70.11 | | -0.50 | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 70.6 | | -0.43 | |
| 2121 | | ----- | | ----- | |
| 2146 | | ----- | | ----- | |
| 2159 | In house | 92.02 | | 2.47 | |
| 2165 | | ----- | | ----- | |
| 2182 | | ----- | | ----- | |
| 2184 | | ----- | | ----- | |
| 2216 | | ----- | | ----- | |
| 2230 | | ----- | | ----- | |
| 2256 | | ----- | | ----- | |
| 2265 | | ----- | | ----- | |
| 2310 | EN16711-1 | 74 | | 0.03 | |
| 2311 | EN16711-1 | 72.11 | | -0.22 | |
| 2314 | EN16711-1 | 75.10 | | 0.18 | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | | ----- | | ----- | |
| 2358 | EN16711-1 | 53.6 | | -2.73 | |
| 2366 | | ----- | | ----- | |
| 2373 | EN16711-1 | 67.2 | | -0.89 | |
| 2375 | EN16711-1 | 74 | | 0.03 | |
| 2380 | EN16711-1 | 64.329 | | -1.28 | |
| 2381 | EN16711-1 | 66.70 | | -0.96 | |
| 2382 | | ----- | | ----- | |
| 2385 | In house | 73.8 | | 0.00 | |
| 2459 | EN16711-1 | 229.76 | R(0.01) | 21.15 | |
| 2475 | | ----- | | ----- | |
| 2500 | | ----- | | ----- | |
| 2503 | CPSC-CH-E1001-08.3 | 85 | | 1.52 | |
| 2511 | EN16711-1 | 80.759 | | 0.95 | |
| 2590 | CPSC-CH-E1001-08.3 | 74.67 | | 0.12 | |
| 2591 | | ----- | | ----- | |
| 2622 | | 74 | | 0.03 | |
| 2624 | | ----- | | ----- | |
| 2637 | | ----- | | ----- | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | CPSC-CH-E1001-08.3 | 82.39 | | 1.17 | |
| 2741 | | ----- | | ----- | |
| 2810 | CPSC-CH-E1001-08.3 | 88.19 | | 1.96 | |
| 2829 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2977 | CPSC-CH-E1001-08.3 | 72.98 | | -0.11 | |
| 2995 | | ----- | | ----- | |
| 3116 | | ----- | | ----- | |
| 3172 | EN16711-1 | 81.3 | | 1.02 | |
| 3209 | | ----- | | ----- | |
| 3214 | EN16711-1 | 66.96 | | -0.92 | |
| 3228 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 22 |
| outliers | 1 |
| mean (n) | 73.766 |
| st.dev. (n) | 8.8030 |
| R(calc.) | 24.648 |
| st.dev.(IEC62321-5:14) | 7.3766 |
| R(IEC62321-5:14) | 20.654 |

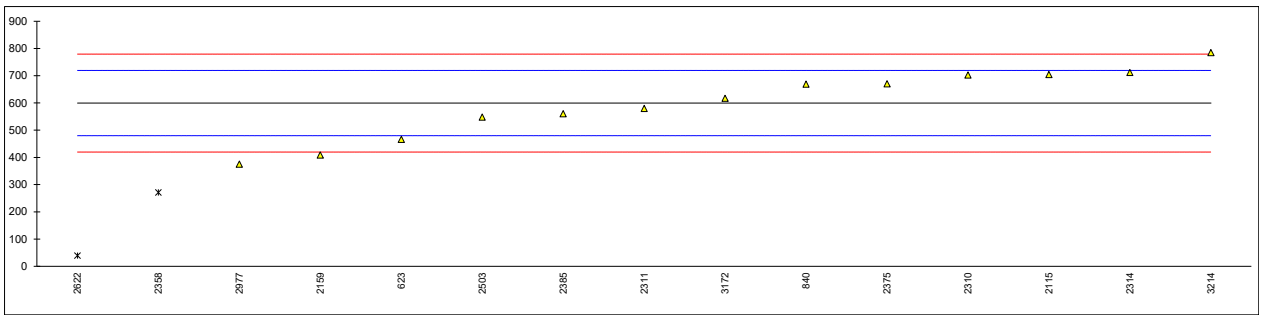

110
100
90
80
70
60
50
40
30
2358
623
2380
2381
3214
2373
840
2115
2311
2977
2385
2375
2310
2622
2590
2314
2511
3172
2737
2503
2810
2159
2459

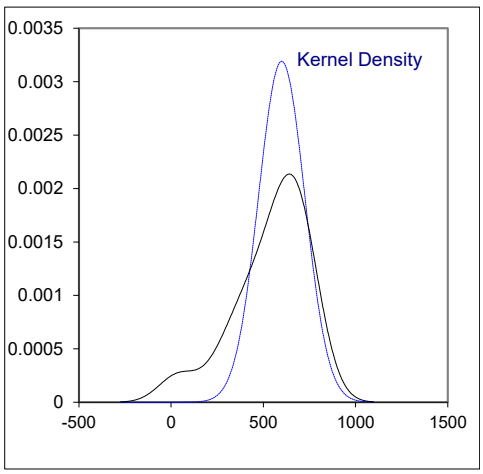

Kernel Density
0.05
0.045
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
0
100
200
300

## Determination of Tin as Sn on sample #23630-2; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 465.98 | | -2.23 | |
| 840 | In house | 668.99 | | 1.16 | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 703.9 | | 1.74 | |
| 2121 | | ----- | | ----- | |
| 2146 | | ----- | | ----- | |
| 2159 | In house | 408.5 | | -3.19 | |
| 2165 | | ----- | | ----- | |
| 2182 | | ----- | | ----- | |
| 2184 | | ----- | | ----- | |
| 2216 | | ----- | | ----- | |
| 2230 | | ----- | | ----- | |
| 2256 | | ----- | | ----- | |
| 2265 | | ----- | | ----- | |
| 2310 | EN16711-1 | 702 | | 1.71 | |
| 2311 | EN16711-1 | 579.11 | | -0.34 | |
| 2314 | EN16711-1 | 711.54 | | 1.87 | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | | ----- | | ----- | |
| 2358 | EN16711-1 | 271 | C,DG(0.05) | -5.48 | first reported 150 |
| 2366 | | ----- | | ----- | |
| 2373 | | ----- | | ----- | |
| 2375 | EN16711-1 | 670 | | 1.17 | |
| 2380 | | ----- | | ----- | |
| 2381 | | ----- | | ----- | |
| 2382 | | ----- | | ----- | |
| 2385 | In house | 560 | | -0.66 | |
| 2459 | | ----- | | ----- | |
| 2475 | | ----- | | ----- | |
| 2500 | | ----- | | ----- | |
| 2503 | CPSC-CH-E1001-08.3 | 548 | | -0.86 | |
| 2511 | | ----- | | ----- | |
| 2590 | | ----- | | ----- | |
| 2591 | | ----- | | ----- | |
| 2622 | | 39 | DG(0.05) | -9.35 | |
| 2624 | | ----- | | ----- | |
| 2637 | | ----- | | ----- | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | | ----- | | ----- | |
| 2741 | | ----- | | ----- | |
| 2810 | | ----- | | ----- | |
| 2829 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2977 | CPSC-CH-E1001-08.3 | 375.0 | | -3.75 | |
| 2995 | | ----- | | ----- | |
| 3116 | | ----- | | ----- | |
| 3172 | EN16711-1 | 617 | | 0.29 | |
| 3209 | | ----- | | ----- | |
| 3214 | EN16711-1 | 784.44 | | 3.08 | |
| 3228 | | ----- | | ----- | |

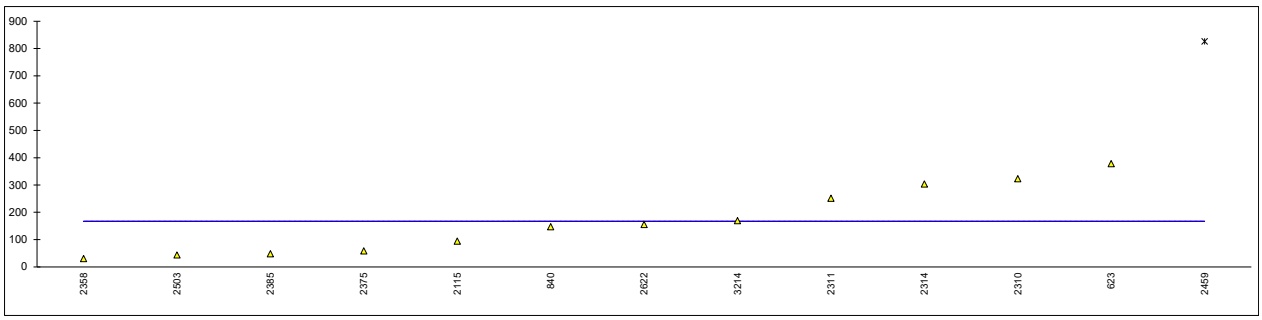
| | |
|---|---|
| normality | OK |
| n | 13 |
| outliers | 2 |
| mean (n) | 599.574 |
| st.dev. (n) | 125.0346 RSD=21% |
| R(calc.) | 350.097 |
| st.dev.(IEC62321-5:14) | 59.9574 |
| R(IEC62321-5:14) | 167.881 |

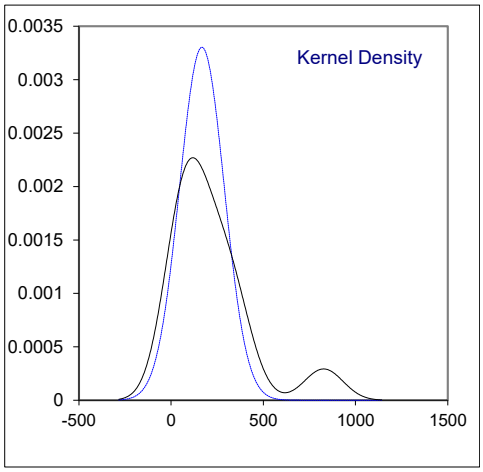

900
800
700
600
500
400
300
200
100
0
2622
2358
2977
2159
623
2503
2385
2311
3172
840
2375
2310
2115
2314
3214


Kernel Density
0.0035
0.003
0.0025
0.002
0.0015
0.001
0.0005
0
-500
0
500
1000
1500

Determination of Zinc as Zn on sample #23630-2; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 210 | | ----- | | ----- | |
| 623 | In house | 377.89 | | ----- | |
| 840 | In house | 146.70 | | ----- | |
| 1126 | | ----- | | ----- | |
| 2115 | EN16711-1 | 94.07 | | ----- | |
| 2121 | | ----- | | ----- | |
| 2146 | | ----- | | ----- | |
| 2159 | | ----- | | ----- | |
| 2165 | | ----- | | ----- | |
| 2182 | | ----- | | ----- | |
| 2184 | | ----- | | ----- | |
| 2216 | | ----- | | ----- | |
| 2230 | | ----- | | ----- | |
| 2256 | | ----- | | ----- | |
| 2265 | | ----- | | ----- | |
| 2310 | EN16711-1 | 323 | | ----- | |
| 2311 | EN16711-1 | 251.36 | | ----- | |
| 2314 | EN16711-1 | 303.47 | | ----- | |
| 2326 | | ----- | | ----- | |
| 2347 | | ----- | | ----- | |
| 2350 | | ----- | | ----- | |
| 2358 | EN16711-1 | 30.3 | | ----- | |
| 2366 | | ----- | | ----- | |
| 2373 | | ----- | | ----- | |
| 2375 | EN16711-1 | 58 | | ----- | |
| 2380 | | ----- | | ----- | |
| 2381 | | ----- | | ----- | |
| 2382 | | ----- | | ----- | |
| 2385 | In house | 48.0 | | ----- | |
| 2459 | EN16711-1 | 826.52 | G(0.01) | ----- | |
| 2475 | | ----- | | ----- | |
| 2500 | | ----- | | ----- | |
| 2503 | CPSC-CH-E1001-08.3 | 43.7 | | ----- | |
| 2511 | | ----- | | ----- | |
| 2590 | | ----- | | ----- | |
| 2591 | | ----- | | ----- | |
| 2622 | | 155 | | ----- | |
| 2624 | | ----- | | ----- | |
| 2637 | | ----- | | ----- | |
| 2678 | | ----- | | ----- | |
| 2734 | | ----- | | ----- | |
| 2737 | | ----- | | ----- | |
| 2741 | | ----- | | ----- | |
| 2810 | | ----- | | ----- | |
| 2829 | | ----- | | ----- | |
| 2864 | | ----- | | ----- | |
| 2977 | | ----- | | ----- | |
| 2995 | | ----- | | ----- | |
| 3116 | | ----- | | ----- | |
| 3172 | | ----- | | ----- | |
| 3209 | | ----- | | ----- | |
| 3214 | EN16711-1 | 169.01 | | ----- | |
| 3228 | | ----- | | ----- | |
| | normality | OK | | | |
| | n | 12 | | | |
| | outliers | 1 | | | |
| | mean (n) | 166.7083 | | | |
| | st.dev. (n) | 120.74050 | RSD=72% | | |
| | R(calc.) | 338.0734 | | | |
| | st.dev.(IEC62321-5:14) | (16.67083) | | | |
| | R(IEC62321-5:14) | (46.6783) | | | |


900
800
700
600
500
400
300
200
100
0
2358
2503
2385
2375
2115
840
2622
3214
2311
2314
2310
623
2459


Kernel Density
0.0035
0.003
0.0025
0.002
0.0015
0.001
0.0005
0
-500
0
500
1000
1500

## APPENDIX 3
## Other reported Metals in sample #23630-1; results in mg/kg

| lab | Sb | As | Cr | Co | Mn |
|---|---|---|---|---|---|
| 210 | ----- | ----- | ----- | ----- | ----- |
| 623 | 75.35 | Not Detected | Not Detected | Not Detected | Not Detected |
| 840 | ----- | ----- | ----- | ----- | ----- |
| 1126 | ----- | ----- | ----- | ----- | ----- |
| 2115 | 1.71 | ----- | ----- | ----- | 1.83 |
| 2121 | ----- | ----- | ----- | ----- | ----- |
| 2146 | ----- | ----- | ----- | ----- | ----- |
| 2159 | ----- | ----- | ----- | ----- | ----- |
| 2165 | ----- | ----- | Not detected | ----- | ----- |
| 2182 | ----- | ----- | ----- | ----- | ----- |
| 2184 | ----- | ----- | <10 | ----- | ----- |
| 2216 | 52 | None Detected | 3 | Not Determined | Not Determined |
| 2230 | ----- | ----- | ----- | ----- | ----- |
| 2256 | ----- | ----- | Not detected | ----- | ----- |
| 2265 | not analysed | not detected | not detected | not analysed | not analysed |
| 2310 | <10 | not detected | not detected | not detected | not detected |
| 2311 | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected |
| 2314 | 9.11 | ----- | ----- | ----- | ----- |
| 2326 | ----- | ----- | ----- | ----- | ----- |
| 2347 | ----- | ----- | ----- | ----- | ----- |
| 2350 | N.A. | N.A. | N.A. | N.A. | N.A. |
| 2358 | not detected | not detected | not detected | not detected | not determined |
| 2366 | ----- | ----- | ----- | ----- | ----- |
| 2373 | not analyzed | not analyzed | not analyzed | not analyzed | not analyzed |
| 2375 | <10 | <10 | <10 | <10 | <10 |
| 2380 | ----- | ----- | ----- | ----- | ----- |
| 2381 | ----- | ----- | ----- | ----- | ----- |
| 2382 | no capability | no capability | no capability | no capability | no capability |
| 2385 | <10 | <10 | <10 | <10 | 128 |
| 2459 | 84.62 | ----- | ND | ND | ND |
| 2475 | ----- | ----- | ----- | ----- | ----- |
| 2500 | ----- | ----- | ND | ND | ND |
| 2503 | 379 | 336 | ----- | ----- | ----- |
| 2511 | ----- | ----- | ----- | ----- | ----- |
| 2590 | < L.O.Q. | ----- | < L.O.Q. | < L.O.Q. | ----- |
| 2591 | ----- | ----- | ----- | ----- | ----- |
| 2622 | ----- | ----- | ----- | ----- | ----- |
| 2624 | ----- | ----- | ----- | ----- | ----- |
| 2637 | 21 | 23 | 150 | 22 | 1790 |
| 2678 | ----- | ----- | ----- | ----- | ----- |
| 2734 | ----- | ----- | ----- | ----- | ----- |
| 2737 | ----- | ----- | ----- | ----- | ----- |
| 2741 | ----- | ----- | ----- | ----- | ----- |
| 2810 | ----- | ----- | ----- | ----- | ----- |
| 2829 | not analyzed | not analyzed | not analyzed | not analyzed | not analyzed |
| 2864 | ----- | ----- | ----- | ----- | ----- |
| 2977 | not detected | not detected | not detected | not detected | not detected |
| 2995 | ----- | ----- | not detected | ----- | ----- |
| 3116 | ----- | ----- | ----- | ----- | ----- |
| 3172 | < 10 | < 10 | < 10 | < 10 | ----- |
| 3209 | ----- | ----- | <10.0 | <10.0 | <10.0 |
| 3214 | <10 | <10 | <10 | <10 | <10 |
| 3228 | ----- | ----- | <10 | ----- | ----- |

## Other reported Metals in sample #23630-1; results in mg/kg -continued

| lab | Hg | Ni | Se | Sr | Zr |
|---|---|---|---|---|---|
| 210 | ----- | ----- | ----- | ----- | ----- |
| 623 | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected |
| 840 | ----- | ----- | ----- | ----- | ----- |
| 1126 | ----- | ----- | ----- | ----- | ----- |
| 2115 | 0.45 | 1.85 | 3.24 | ----- | ----- |
| 2121 | ----- | ----- | ----- | ----- | ----- |
| 2146 | ----- | ----- | ----- | ----- | ----- |
| 2159 | ----- | ----- | ----- | ----- | ----- |
| 2165 | Not detected | ----- | ----- | ----- | ----- |
| 2182 | ----- | ----- | ----- | ----- | ----- |
| 2184 | <10 | ----- | ----- | ----- | ----- |
| 2216 | None Detected | Not Determined | None Detected | Not Determined | Not Determined |
| 2230 | ----- | ----- | ----- | ----- | ----- |
| 2256 | Not detected | ----- | ----- | ----- | ----- |
| 2265 | not detected | not analysed | not analysed | not analysed | not analysed |
| 2310 | not detected | not detected | not detected | not detected | not detected |
| 2311 | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected |
| 2314 | ----- | ----- | ----- | ----- | ----- |
| 2326 | ----- | ----- | ----- | ----- | ----- |
| 2347 | ----- | ----- | ----- | ----- | ----- |
| 2350 | < 2 | N.A. | N.A. | N.A. | N.A. |
| 2358 | not detected | not detected | not detected | not determined | not determined |
| 2366 | ----- | ----- | ----- | ----- | ----- |
| 2373 | not analyzed | not analyzed | not analyzed | not analyzed | not analyzed |
| 2375 | <10 | <10 | <10 | <10 | <10 |
| 2380 | ----- | ----- | ----- | ----- | ----- |
| 2381 | ----- | ----- | ----- | ----- | ----- |
| 2382 | no capability | no capability | no capability | no capability | no capability |
| 2385 | <1 | <10 | <10 | <10 | <10 |
| 2459 | ----- | ND | ND | ND | ----- |
| 2475 | ----- | ----- | ----- | ----- | ----- |
| 2500 | ND | ----- | ----- | ----- | ----- |
| 2503 | ----- | 41.7 | 508 | ----- | ----- |
| 2511 | ----- | ----- | ----- | ----- | ----- |
| 2590 | < L.O.Q. | < L.O.Q. | ----- | < L.O.Q. | ----- |
| 2591 | ----- | ----- | ----- | ----- | ----- |
| 2622 | ----- | 212 | ----- | ----- | ----- |
| 2624 | ----- | ----- | ----- | ----- | ----- |
| 2637 | not detected | 45 | not detected | not detected | not detected |
| 2678 | ----- | ----- | ----- | ----- | ----- |
| 2734 | ----- | ----- | ----- | ----- | ----- |
| 2737 | ----- | ----- | ----- | ----- | ----- |
| 2741 | ----- | ----- | ----- | ----- | ----- |
| 2810 | ----- | ----- | ----- | ----- | ----- |
| 2829 | not analyzed | not analyzed | not analyzed | not analyzed | not analyzed |
| 2864 | not detected | ----- | ----- | ----- | ----- |
| 2977 | not detected | not detected | not detected | not detected | not detected |
| 2995 | not detected | ----- | ----- | ----- | ----- |
| 3116 | ----- | ----- | ----- | ----- | ----- |
| 3172 | < 10 | < 10 | ----- | ----- | ----- |
| 3209 | <5.0 | ----- | ----- | ----- | ----- |
| 3214 | <10 | <10 | <10 | <10 | <10 |
| 3228 | <10 | ----- | ----- | ----- | ----- |

## Other reported Metals in sample #23630-2; results in mg/kg

| lab | Sb | Cd | Pb | Hg | Se | Sr | Zr |
|---|---|---|---|---|---|---|---|
| 210 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 623 | Not Detected | 15.69 | 30.04 | Not Detected | Not Detected | Not Detected | Not Detected |
| 840 | <10 | <10 | <10 | <10 | <10 | <10 | not analyzed |
| 1126 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2115 | 2.98 | 1.98 | 1.59 | ----- | ----- | ----- | ----- |
| 2121 | ----- | ----- | 0.8825 | ----- | ----- | ----- | ----- |
| 2146 | ----- | 33.5 | < 100 mg/kg | ----- | ----- | ----- | ----- |
| 2159 | Not applicable | Not applicable | Not applicable | Not applicable | Not applicable | Not applicable | Not applicable |
| 2165 | ----- | Not detected | Not detected | Not detected | ----- | ----- | ----- |
| 2182 | ----- | not detected | not detected | ----- | ----- | ----- | ----- |
| 2184 | ----- | <10 | <10 | <10 | ----- | ----- | ----- |
| 2216 | None Detected | 20 | 2 | None Detected | None Detected | Not Determined | Not Determined |
| 2230 | ----- | ----- | <10 | ----- | ----- | ----- | ----- |
| 2256 | ----- | Not detected | ND | Not detected | ----- | ----- | ----- |
| 2265 | not analysed | not detected | not detected | not detected | not analysed | not analysed | not analysed |
| 2310 | not detected | not detected | 38.2 | not detected | not detected | not detected | not detected |
| 2311 | Not Detected | Not Detected | 28.38 | Not Detected | Not Detected | Not Detected | Not Detected |
| 2314 | ----- | ----- | 48.73 | ----- | ----- | ----- | ----- |
| 2326 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2347 | ----- | <5 | <20 | ----- | ----- | ----- | ----- |
| 2350 | N.A. | < 5 | < 50 | < 2 | N.A. | N.A. | N.A. |
| 2358 | not detected | not detected | not detected | not detected | not detected | not determined | not determined |
| 2366 | <20 | <5 | <10 | <5 | <20 | ----- | ----- |
| 2373 | ＜20 | ＜5 | ＜20 | <5 | not applicable | not applicable | not applicable |
| 2375 | <10 | <10 | <10 | <10 | <10 | <10 | <10 |
| 2380 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2381 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2382 | no capability | no capability | not detected | no capability | no capability | no capability | no capability |
| 2385 | <10 | <10 | <20 | <1 | <10 | <10 | <10 |
| 2459 | 310.01 | 28.21 | 98.42 | ----- | ----- | 198.00 | ----- |
| 2475 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2500 | ----- | ND | ND | ND | ----- | ----- | ----- |
| 2503 | 35 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2511 | ----- | ----- | <10 | ----- | ----- | ----- | ----- |
| 2590 | < L.O.Q. | 2.10 | < L.O.Q. | < L.O.Q. | ----- | < L.O.Q. | ----- |
| 2591 | ----- | not detected | not detected | ----- | ----- | ----- | ----- |
| 2622 | ----- | <5 | <25 | ----- | ----- | ----- | ----- |
| 2624 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2637 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2678 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2734 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2737 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2741 | ----- | ----- | <20 | ----- | ----- | ----- | ----- |
| 2810 | ----- | not detected | not detected | ----- | ----- | ----- | ----- |
| 2829 | not analyzed | not detected | not detected | not analyzed | not analyzed | not analyzed | not analyzed |
| 2864 | ----- | 41.26 | not detected | not detected | ----- | ----- | ----- |
| 2977 | not detected | not detected | not detected | not detected | not detected | not detected | not detected |
| 2995 | ----- | not detected | not detected | not detected | ----- | ----- | ----- |
| 3116 | ----- | ----- | <50 | ----- | ----- | ----- | ----- |
| 3172 | < 10 | 10.6 | < 5 | < 10 | ----- | ----- | ----- |
| 3209 | ----- | <5.0 | <10.0 | <5.0 | ----- | ----- | ----- |
| 3214 | <10 | <10 | <10 | <10 | <10 | <10 | <10 |
| 3228 | ----- | <10 | <10 | <10 | ----- | ----- | ----- |

## **APPENDIX 4** Analytical details

| lab | Accredited ISO17025 | Intake in grams | Digestion acid used | Concentration Acid | Quantify technique used |
|---|---|---|---|---|---|
| 210 | --- | | | | --- |
| 623 | Yes | 0.2 | HNO3 Aquaregia for Sn | 16 | ICP-OES |
| 840 | Yes | 0.3 | 7.5mLHNO3:2.5mLHCl | 14% | ICP-OES and ICP-MS |
| 1126 | --- | | | | --- |
| 2115 | Yes | 0.1 g | HNO3 | | ICP-MS |
| 2121 | Yes | 80mg | 4.5mL HNO3 + 1.5mL HCl | HNO3 69.5% HCl 37% | ICP-MS |
| 2146 | No | #23630-1: whole sample (1,9 g) was digested #23630-2: 0,2 g | HNO3 | 67 % (wt/wt) | ICP-OES |
| 2159 | Yes | 0.2 grams | Nitric acid | % 65 nitric acid | ICP-OES |
| 2165 | Yes | 0.1g, nearest to 0.1mg | Aqua regia | 8%(V/V) Aqua regia | ICP-OES |
| 2182 | Yes | 0.1g | Nitric acid | 67-69% | AAS |
| 2184 | Yes | 0.1g | HNO3 and HCl | 24% | ICP-MS |
| 2216 | Yes | #23630-1 = 0.9758 g #23630-2 = 0.98 g | Nitric and hydrochloric acids | 9% HNO3 / 6% HCl | ICP-MS |
| 2230 | Yes | 0.2388 g | aqua regia | 40% | ICP-OES |
| 2256 | Yes | 23630-1:0.0726g 23630-2:0.0656g | conc. HNO3 & conc. HCl | Acid mixture (10% nitric acid + 4% hydrochloric acid) | ICP-OES |
| 2265 | Yes | 0,1 | HNO3 / HCl | 3 / 1 | ICP-OES |
| 2310 | Yes | 0.25 | Conc.Nitric acid | 69% | ICP-MS |
| 2311 | Yes | 0.1 | Nitric Acid | 69 | ICP-OES |
| 2314 | Yes | 0.1g | Nitric acid | 69% | ICP-OES |
| 2326 | --- | | | | --- |
| 2347 | No | 0.25 | HNO3/HCl | 20% | ICP-OES |
| 2350 | Yes | approximately 0.2g | Nitric acid & hydrochloric acid | Nitric acid 70% , hydrochloric acid 36% | ICP-OES |
| 2358 | Yes | 0.25 | HNO3 | 65 | ICP-MS |
| 2366 | Yes | | | | --- |
| 2373 | Yes | 0.1g | HCL,HNO3 | HCL:36%~38% HNO3:65%~68% | ICP-OES |
| 2375 | Yes | 0.15 gram | HNO3 + HCl | 9 mL + 3 mL | ICP-MS |
| 2380 | Yes | 0.20 g | HNO3 & HCl | HNO3-65% & HCl-37% | ICP-OES |
| 2381 | Yes | 0.25 | Nitric acid | 65% | ICP-OES |
| 2382 | Yes | 1.5g | HNO3&HCL | HNO3：69.0~72.0% HCL：36.0~38.0% | AAS |
| 2385 | --- | | | | --- |
| 2459 | Yes | 0.2 | Aqua Regia | Conc. Acids (Nitric and Hydro Chloric Acid is used for digestion) | ICP-OES / AAS |
| 2475 | No | 0.1 | 2.5 ml HNO3 and 7.5 ml HCl | | ICP-MS |
| 2500 | Yes | 0.1 gram | Nitric acid / Hydrochloric acid | Nitric acid 65-70% Hydrochloric acid 37% | ICP-OES |
| 2503 | --- | | | | --- |
| 2511 | --- | | | | --- |
| 2590 | Yes | 0.2 gr | HNO3 | 4% | ICP-MS |
| 2591 | Yes | 0,1 grams | Nitric Acid | | ICP-MS |
| 2622 | Yes | 0.5117 gr | Nitric acid and hydrochloric acid | | AAS |
| 2624 | Yes | 0.1506 | HNO3 | ~ 20% | ICP-OES |
| 2637 | No | | | | ICP-MS |
| 2678 | --- | | | | --- |
| 2734 | --- | | | | --- |
| 2737 | Yes | 0.2g | HNO3:HCl=1:3 | HNO3:65% HCl：36% | ICP-OES |
| 2741 | Yes | 0.1g | HNO3 65%. | 3% | ICP-MS |
| 2810 | Yes | 0,8577g sample 1 1,4266g sample 2 | Nitric acid / Hydrochloric acid | 37% Hydrochloric acid / 65% Nitric acid | ICP-OES |
| 2829 | No | 0.09 | Nitric and hydrochloric acid | HCl 37% and HNO3 65% | ICP-OES |
| 2864 | No | 0.2g | Nitric acid | 65% | ICP-OES |
| 2977 | Yes | 0,1 | HNO3 HCl | 7 | ICP-OES |
| 2995 | No | #22630-1: 0.9494 g #22630-2: 1.0259 g | HNO3 + HCl | HNO3 65% / HCl 37% | ICP-OES |
| 3116 | Yes | 0.1 (for both CPSC and Canada methods) | Conc. nitric acid and conc. hydrochloric acid (for both CPSC & Canada methods) | 8 mL HNO3 / 3 mL HCl (for CPSC method) 8 mL HNO3 / 2 mL HCl (for Canada method) | ICP-OES |
| 3172 | Yes | | | | --- |
| 3209 | Yes | | aqua regia ( hydrochloric acid ; Nitric acid = 3:1 V/V ) | 20% | ICP-OES |
| 3214 | No | 0.15 grams | 9ml Hcl, 3ml HNO3 1 ml HF | 26% | ICP-OES |
| 3228 | Yes | 0.1-0.2g | concentrated HNO3 and HCl | 10%HNO3+10%HCl in sample solution | ICP-OES |

## APPENDIX 5

## Number of participants per country

2 labs in BANGLADESH
1 lab in FINLAND
2 labs in FRANCE
3 labs in GERMANY
4 labs in HONG KONG
3 labs in INDIA
1 lab in INDONESIA
9 labs in ITALY
1 lab in KOREA, Republic of
1 lab in MOROCCO
10 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in SPAIN
3 labs in TAIWAN
1 lab in THE NETHERLANDS
3 labs in TUNISIA
2 labs in TURKEY
2 labs in U.S.A.
2 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 |
| 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 |

### Literature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11 W. Horwitz and R. Albert, J. AOAC Int, 79, 3, 589-621, (1996)
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