

**Results of Proficiency Test  
Total Metals in Metal / Metal Alloy  
June 2021**

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

**Author:** ing. R.J. Starink

**Correctors:** ing. C.M. Nijssen-Wester & ing. A.S. Noordman-de Neef

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## 1 INTRODUCTION

Heavy metals can be found in everything from jewelry and watch cases to electronic components and toy paints. 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 doesn't 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 used to make jewelry in order to impart specific technical and functional attributes to the metal. 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.

The determination of Metals in Metal is known to give problems with the comparability of laboratory results. However, no appropriate Metals reference materials are yet available. As an alternative, participation in a proficiency test may enable laboratories to check their performance. Therefore, a proficiency test (laboratory-evaluating interlaboratory study) for the determination of Metals in Metal was organized by the Institute for Interlaboratory Studies in June 2021 on request of many participants.

In this new interlaboratory study 57 laboratories in 26 different countries registered for participation. See appendix 8 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](http://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 were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send 1 metal necklace labelled #21630.

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](http://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 necklaces, which was recalled from the market, was selected. Each necklace was packed separately and labelled #21630.

The homogeneity of the subsamples (main chain) was checked by determination of the Lead according to an in house method on 5 stratified randomly selected subsamples.

	Lead in mg/kg
sample #21630-1	19.0
sample #21630-2	19.7
sample #21630-3	18.7
sample #21630-4	19.0
sample #21630-5	19.3

Table 1: homogeneity test results of subsamples #21630

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility calculated with the Horwitz equation in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Lead in mg/kg
r (observed)	1.1
reference method	Horwitz
0.3 x R (reference method)	1.6

Table 2: evaluation of the repeatability of subsamples #21630

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample #21630 was sent on May 19, 2021.

## 2.5 ANALYZES

The participants were requested to determine: 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 and Zinc as Zn.

It was also requested to report if the laboratory was accredited for the requested elements that were determined 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.

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/](http://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](http://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/](http://www.kpmd.co.uk/sgs-iis-cts/). The reported test results are tabulated per determination in appendices 1 to 6 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 re-analysis). 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 4. 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' (iis-protocol, version 3.5) of June 2018. 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). 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, 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:

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

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

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

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

## 4 EVALUATION

In this interlaboratory study some problems were encountered. After receipt of the samples several participants requested iis how to analyze the necklace and how to report their findings as there was only place for one set on the data entry portal. The participants mentioned that the necklace could be split into two, three or four different parts. It was decided to create four identical forms on the data entry portal. The participants were requested to mention the part analyzed on each form.

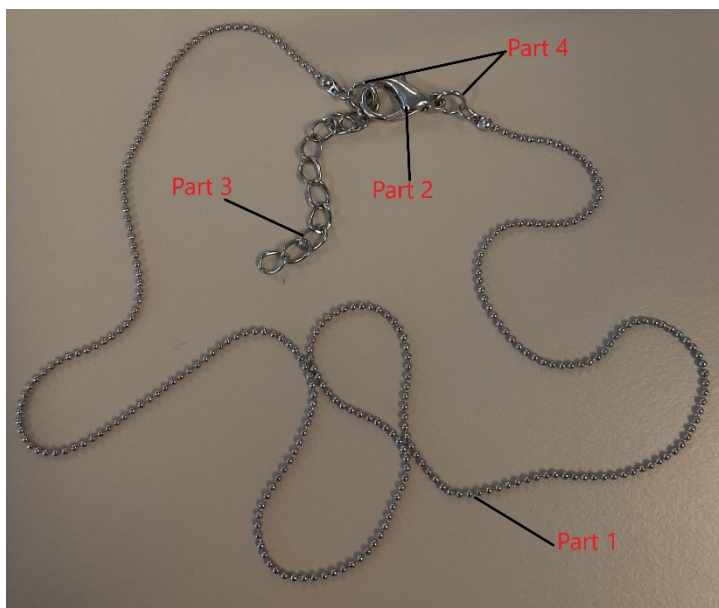
Seven participants reported the test results after the final reporting date and one participant did not report any test results. Not all participants were able to report all elements requested. Finally, 56 reporting laboratories submitted 350 numerical test results. Observed were 26 outlying test results, which is 7.4%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original 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.

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

In this section the results are discussed per part of the necklace 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 appendix 1. The abbreviations, used in these tables, are explained in appendix 9.

The test results reported by the participants the necklace could be divided into 4 parts. The test results were listed in appendix 1 to 4 as follows:



Part 1 (#21630-1): Main Chain (balls)

Part 2 (#21630-2): Clasp (lobster claw)

Part 3 (#21630-3): Extension rings

Part 4 (#21630-4): Large rings (connection between main chain and closure)

Test method CPSC-CH-E1001.08 (Determination of Total Lead in Metal Products) may be considered to be the official test method for the determination of Metals in Metal/ Metal Alloys. Unfortunately, there is no precision data mentioned in CPSC-CH-E1001.08 and there is no other test method available with usable precision data. Therefore, the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

##### **Sample #21630-1 (Main Chain, small balls)**

Cadmium as Cd: This determination may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

Copper as Cu: This determination may be problematic. One statistical outlier was observed. The consensus value was high (67%M/M). A large variation in the reported test results is found, therefore no z-scores are calculated.



- Lead as Pb: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.
- Nickel as Ni: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated with the Horwitz equation.
- Zinc as Zn: This determination may be problematic. One statistical outlier was observed. The consensus value was high (33%M/M). A large variation in the reported test results is found, therefore no z-scores are calculated.
- Other metals: The majority of participants agreed on a concentration near or below the limit of detection for the other elements. The test results are given in appendix 6.

**Sample #21630-2 (Clasp, lobster claw)**

- Cadmium as Cd: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.
- Copper as Cu: This determination may be problematic. One statistical outlier was observed. The consensus value was high (1%M/M). A large variation in the reported test results is found, therefore no z-scores are calculated.
- Lead as Pb: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated with the Horwitz equation.
- Zinc as Zn: This determination may be problematic. Three statistical outliers were observed. The consensus value was high (93%M/M). A large variation in the reported test results is found, therefore no z-scores are calculated.
- Other metals: The majority of participants agreed on a concentration near or below the limit of detection for the other elements. The test results are given in appendix 6.

**Sample #21630-3 (Extension rings)**

- Arsenic as As: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.
- Chromium as Cr: This determination may be problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

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

Copper as Cu: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

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

Nickel as Ni: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

Zinc as Zn: This determination may be problematic. Two statistical outliers were observed. A large variation in the reported test results is found, therefore no z-scores are calculated.

Other metals: The majority of participants agreed on a concentration near or below the limit of detection for the other elements. The test results are given in appendix 6.

#### **Sample #21630-4 (Larger rings)**

Chromium as Cr: This determination may be problematic. Two statistical outliers were observed. A large variation in the reported test results is found, therefore no z-scores are calculated.

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

Lead as Pb: This determination may be problematic. One statistical outlier was observed. The consensus value was high (1%M/M). A large variation in the reported test results is found, 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 full agreement with the estimated reproducibility calculated with the Horwitz equation.

Nickel as Ni: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated reproducibility calculated with the Horwitz equation.

**Zinc as Zn:** This determination may be problematic. No statistical outliers were observed. A large variation in the reported test results is found, therefore no z-scores are calculated.

**Other metals:** The majority of participants agreed on a concentration near or below the limit of detection for the other elements. The test results are given in appendix 6.

#### **Sample #21630 (not identified parts of the necklace)**

Six participants reported a variety of test results which could not be identified to a part of the necklace. These test results are given in appendix 5 and could unfortunately not be further evaluated.

## **4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES**

A comparison has been made between the reproducibility as declared by the estimated target reproducibility calculated with the Horwitz equation 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 estimated calculated with the Horwitz equation are presented in the next table.

Element	unit	n	average	2.8 * sd	R(target)
Cadmium as Cd	mg/kg	31	10.0	4.7	3.2
Copper as Cu	mg/kg	15	670000	190000	(40000)
Lead as Pb	mg/kg	35	20.4	13.9	5.8
Nickel as Ni	mg/kg	14	24.0	7.8	6.7
Zinc as Zn	mg/kg	13	330000	90000	(22000)

Table 3: performance overview on sample #21630-1 (main ball chain)

Element	unit	n	average	2.8 * sd	R(target)
Cadmium as Cd	mg/kg	10	4.5	1.9	1.6
Copper as Cu	mg/kg	13	9700	11450	(1090)
Lead as Pb	mg/kg	41	278	193	53
Zinc as Zn	mg/kg	8	930000	180000	(53000)

Table 4: performance overview on sample #21630-2 (clasp)

Element	unit	n	average	2.8 * sd	R(target)
Arsenic as As	mg/kg	13	68.6	39.1	16.3
Chromium as Cr	mg/kg	17	220	90	44
Cobalt as Co	mg/kg	12	45.4	25.4	11.5
Copper as Cu	mg/kg	13	13400	4935	1435
Manganese as Mn	mg/kg	13	2759	669	375
Nickel as Ni	mg/kg	15	123	38	27
Zinc as Zn	mg/kg	9	125	155	(27)

Table 5: performance overview on sample #21630-3 (extension rings)

Element	unit	n	average	2.8 * sd	R(target)
Chromium as Cr	mg/kg	11	85.5	61.4	(19.6)
Copper as Cu	mg/kg	8	10050	4040	(1120)
Lead as Pb	mg/kg	10	33.9	52.8	(8.9)
Manganese as Mn	mg/kg	8	3039	404	407
Nickel as Ni	mg/kg	8	39.1	7.3	10.1
Zinc as Zn	mg/kg	7	78.3	184.2	(18.2)

Table 6: performance overview on sample #21630-4 (large connection rings)

Without further statistical calculations, it can be concluded that there is not a good compliance of the group of participating laboratories with the reference method. See also the discussion in paragraphs 4.1 and 5.

### 4.3 OVERVIEW OF THE PROFICIENCY TEST OF JUNE 2021

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

Element	June 2021
Arsenic as As	20%
Cadmium as Cd	15 – 17%
Chromium as Cr	15 – 26%
Cobalt as Co	20%
Copper as Cu	10 – 42%
Lead as Pb	24 – 56%
Manganese as Mn	5 – 9%
Nickel as Ni	7 – 12%
Zinc as Zn	7 – 84%

Table 7: development of uncertainties in % of Metals in Metal

### 4.4 EVALUATION OF THE ANALYTICAL DETAILS

The participants were asked to provide several analytical details which are listed in appendix 7. Based on the reported answers the following can be summarized:

- Forty participants reported to be ISO/IEC17025 accredited to determine the reported elements for Metals in Metal/ Metal Alloys.
- A majority of the laboratories that reported analytical details mentioned to have used Aqua Regia (a mixture of concentrated Nitric acid and concentrated Hydrochloric acid) as digestion acid. A number of other laboratories mentioned to have used only Nitric acid (in different concentrations).
- Almost 90% used 0.5 grams or less as sample intake, most times 0.1 to 0.2 grams.
- The technique to quantify the metals was most often ICP-OES or ICP-MS followed by AAS. One participant reported to have used XRF.

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

## **5 DISCUSSION**

In this proficiency test for the determination of Metals in Metal/ Metal Alloys a necklace was used. Surprisingly, the necklace contained four different parts each with a different metal alloy composition.

The necklace was recalled from the local market and obtained from a local supplier. The recall was made on a high concentration of Lead. It turned out that the clasp contained a high level of Lead.

The large variations found in the determinations of metals for each part of the necklace may partly be explained by the small number of reported test results for each element.

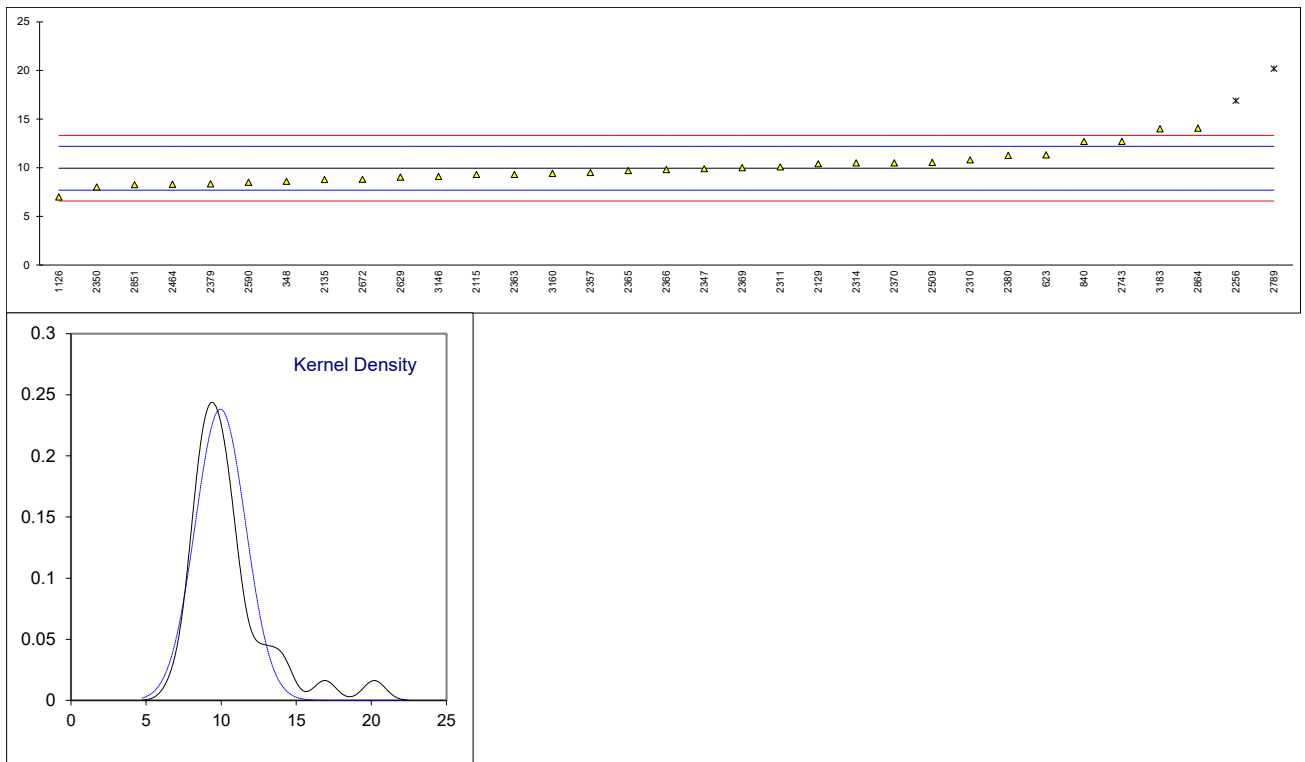
## **6 CONCLUSION**

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 – MAIN CHAIN (small balls)**

Determination of Cadmium as Cd on sample #21630-1; results in mg/kg

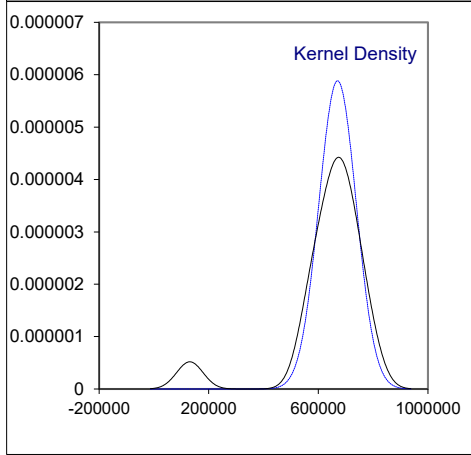
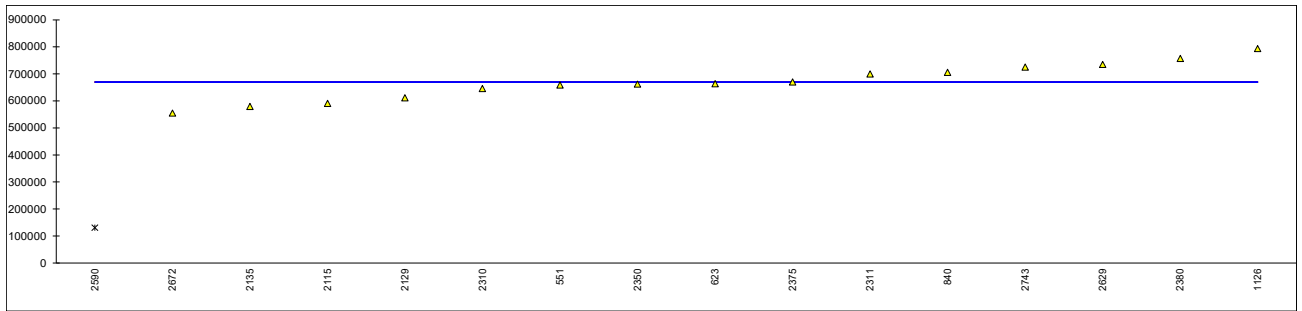
lab	method	value	mark	z(targ)	remarks
110	In house	Not detected		----	
210		----		----	
348	In house	8.6		-1.20	
551		----		----	
623	In house	11.31		1.21	
840	In house	12.69	C	2.43	First reported 15.88
841		----		----	
1126	In house	7.0		-2.62	
1910	In house	not detected		----	
2115	EN16711-1	9.3		-0.58	
2121		----		----	
2129	In house	10.41		0.41	
2135		8.783		-1.04	
2159	In house	<10		----	
2184	CPSC-CH-E1001-08.3	<10		----	
2216		----		----	
2256	CPSC-CH-E1001-08.3	16.9	R(0.05)	6.17	
2266		----		----	
2310	CPSC-CH-E1001-08.3	10.8		0.75	
2311	EN16711-1	10.08		0.11	
2314	In house	10.5		0.49	
2330		----		----	
2347	In house	9.9		-0.05	
2350	EPA3052 / IEC62321-4,5	8.008		-1.73	
2357	In house	9.5		-0.40	
2363	C02.4	9.3		-0.58	
2365	In house	9.702		-0.22	
2366	C02.4.1	9.8		-0.13	
2369		10.0		0.04	
2370	EPA3052 / IEC62321-4,5	10.5		0.49	
2375	ISO16711-1	<10		----	
2379	IEC62321-4,5	8.34		-1.43	
2380	CPSC-CH-E1001-08.3	11.275		1.17	
2385		----		----	
2406	ASTM F963-17	not detected		----	
2464	CPSC-CH-E1001-08.3	8.3		-1.47	
2476		----		----	
2492		----		----	
2509	CPSC-CH-E1001-08	10.5496983		0.53	
2553		----		----	
2564		not detected		----	
2590	EN16711-1	8.50		-1.29	
2624		----		----	
2629	In house	9.04		-0.81	
2656	In house	not detected		----	
2672	DIN13094-3	8.805		-1.02	
2678		----		----	
2703		----		----	
2743		12.70032		2.44	
2789	CPSC-CH-E1001-08.3	20.19	R(0.01)	9.09	
2810	CPSC-CH-E1001-08.3	----		----	
2829		----		----	
2851		8.2429		-1.52	
2864	In house	14.06		3.65	
3146	ISO17294-2 / ISO17852-4	9.098		-0.76	
3160	In house	9.41		-0.48	
3183		14		3.59	
	normality	OK			
	n	31			
	outliers	2			
	mean (n)	9.9517			
	st.dev. (n)	1.67501	RSD = 17%		
	R(calc.)	4.6900			
	st.dev.(Horwitz)	1.12673			
	R(Horwitz)	3.1548			



Determination of Copper as Cu on sample #21630-1; results in mg/kg

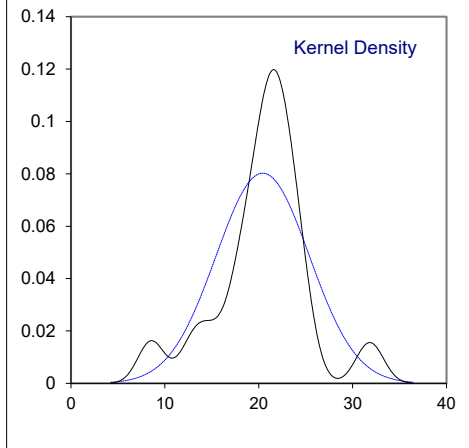
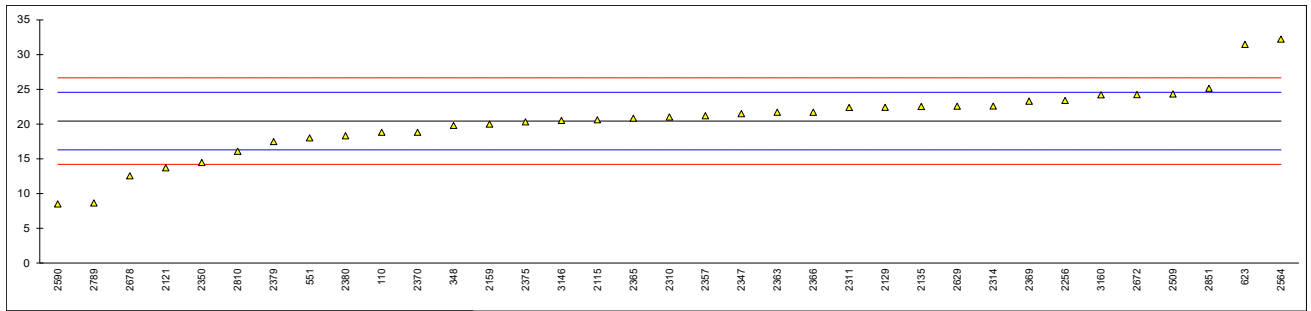
lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		658800		----	
623	In house	663701.45		----	
840	In house	705707		----	
841		----		----	
1126	In house	793600		----	
1910		----		----	
2115	EN16711-1	590525		----	
2121		----		----	
2129	In house	611163		----	
2135		579239		----	
2159		----		----	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	645542		----	
2311	EN16711-1	699341.5		----	
2314		----		----	
2330		----		----	
2347		----		----	
2350	EPA3052 / IEC62321-4,5	662100		----	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375	ISO16711-1	670162		----	
2379		----		----	
2380	CPSC-CH-E1001-08.3	757195		----	
2385		----		----	
2406		----		----	
2464		----		----	
2476		----		----	
2492		----		----	
2509		----		----	
2553		----		----	
2564		----		----	
2590	EN16711-1	130508.30	G(0.01)	----	
2624		----		----	
2629	In house	735100		----	
2656	In house	detected		----	
2672	DIN13094-3	554800		----	
2678		----		----	
2703		----		----	
2743		725014.423	C	----	First reported 725.0144
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146		----		----	
3160		----		----	
3183		----		----	
	normality	OK			
	n	15			
	outliers	1			
	mean (n)	670132.75			
	st.dev. (n)	67819.398	RSD = 10%		
	R(calc.)	189894.32			
	st.dev.(Horwitz)	(14234.960)			
	R(Horwitz)	(39857.89)			





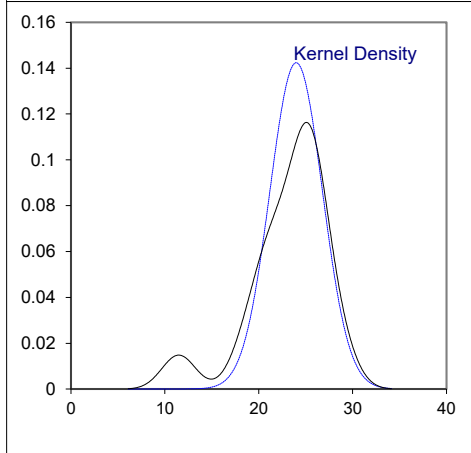
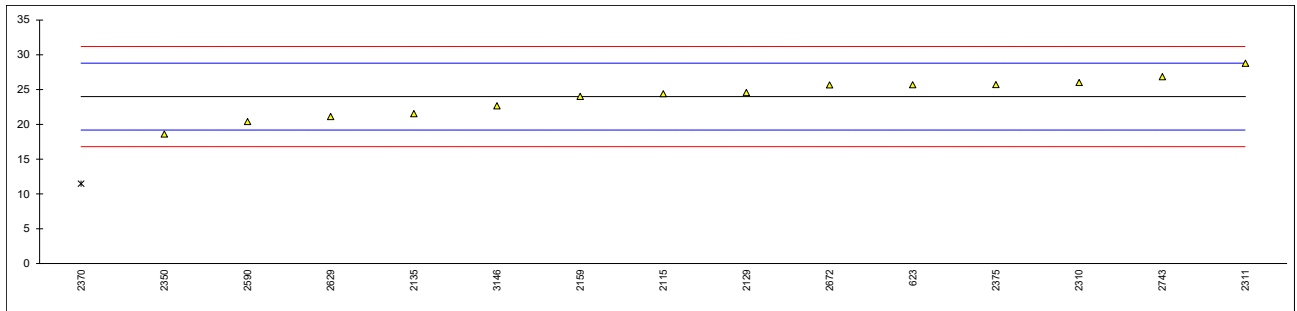
Determination of Lead as Pb on sample #21630-1; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	18.8		-0.79	
210		----		----	
348	In house	19.8		-0.31	
551		18		-1.17	
623	In house	31.47		5.31	
840	In house	<10		<-5.03	Possibly a false negative test result?
841		----		----	
1126		----		----	
1910	In house	not detected		----	
2115	EN16711-1	20.61		0.08	
2121		13.7		-3.24	
2129	In house	22.40		0.95	
2135		22.52		1.00	
2159	In house	20		-0.21	
2184	CPSC-CH-E1001-08.3	<10		<-5.03	Possibly a false negative test result?
2216		----		----	
2256	CPSC-CH-E1001-08.3	23.4		1.43	
2266		----		----	
2310	CPSC-CH-E1001-08.3	21		0.27	
2311	EN16711-1	22.39		0.94	
2314	In house	22.6		1.04	
2330		----		----	
2347	In house	21.5		0.51	
2350	EPA3052 / IEC62321-4,5	14.47		-2.87	
2357	In house	21.2		0.37	
2363	C02.4	21.7		0.61	
2365	In house	20.83		0.19	
2366	C02.4.1	21.7		0.61	
2369		23.3		1.38	
2370	EPA3052 / IEC62321-4,5	18.8		-0.79	
2375	ISO16711-1	20.3		-0.07	
2379	IEC62321-4,5	17.48		-1.42	
2380	CPSC-CH-E1001-08.3	18.318		-1.02	
2385		----		----	
2406	ASTM F963-17	not detected		----	
2464		----		----	
2476		----		----	
2492		----		----	
2509	CPSC-CH-E1001-08	24.3164729		1.87	
2553		----		----	
2564		32.220		5.68	
2590	EN16711-1	8.50		-5.75	
2624		----		----	
2629	In house	22.58		1.03	
2656	In house	not detected		----	
2672	DIN13094-3	24.25		1.84	
2678	CPSC-CH-E1001-08.3	12.54		-3.80	
2703		----		----	
2743		----		----	
2789	CPSC-CH-E1001-08.3	8.62		-5.69	
2810	CPSC-CH-E1001-08.3	16.075		-2.10	
2829		----		----	
2851		25.1533		2.27	
2864		----		----	
3146	ISO17294-2 / ISO17852-4	20.51		0.04	
3160	In house	24.19		1.81	
3183		<LOQ		----	
	normality	suspect			
	n	35			
	outliers	0			
	mean (n)	20.4355			
	st.dev. (n)	4.96994	RSD = 24%		
	R(calc.)	13.9158			
	st.dev.(Horwitz)	2.07622			
	R(Horwitz)	5.8134			



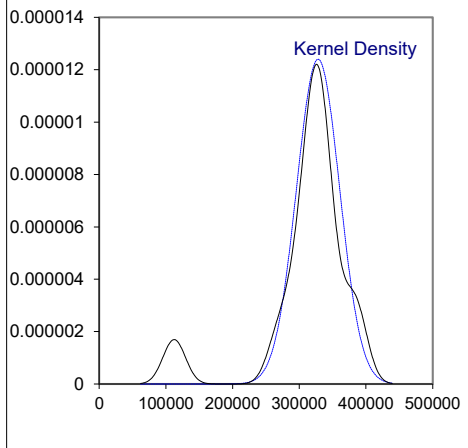
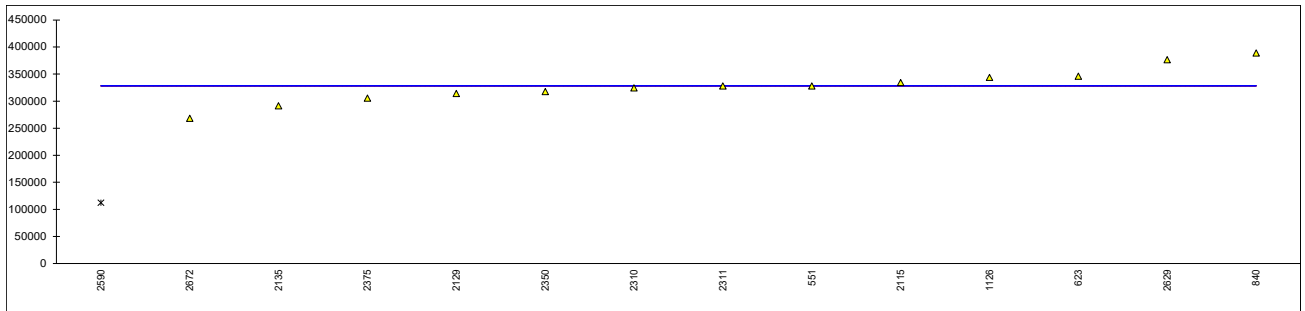
Determination of Nickel as Ni on sample #21630-1; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	25.68		0.71	
840	In house	<10		<-5.88	Possibly a false negative test result?
841		----		----	
1126		----		----	
1910		----		----	
2115	EN16711-1	24.4		0.17	
2121		----		----	
2129	In house	24.55		0.23	
2135		21.54		-1.03	
2159	In house	24		0.00	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	26		0.84	
2311	EN16711-1	28.767		2.01	
2314		----		----	
2330		----		----	
2347		----		----	
2350	EPA3052 / IEC62321-4,5	18.59		-2.27	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370	EPA3052 / IEC62321-4,5	11.473	C,G(0.05)	-5.26	First reported <10
2375	ISO16711-1	25.7		0.72	
2379		----		----	
2380		----		----	
2385		----		----	
2406		----		----	
2464		----	W	----	Test result withdrawn, reported 39.45
2476		----		----	
2492		----		----	
2509		----		----	
2553		----		----	
2564		----		----	
2590	EN16711-1	20.40		-1.51	
2624		----		----	
2629	In house	21.11		-1.21	
2656	In house	not detected		----	
2672	DIN13094-3	25.65		0.70	
2678		----		----	
2703		----		----	
2743		26.83574		1.20	
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146	ISO17294-2 / ISO17852-4	22.66		-0.56	
3160		----		----	
3183		----		----	
	normality	OK			
	n	14			
	outliers	1			
	mean (n)	23.9916			
	st.dev. (n)	2.80233	RSD = 12%		
	R(calc.)	7.8465			
	st.dev.(Horwitz)	2.37936			
	R(Horwitz)	6.6622			



Determination of Zinc as Zn on sample #21630-1; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		328000		----	
623	In house	346039.21		----	
840	In house	389030		----	
841		----		----	
1126	In house	343800		----	
1910		----		----	
2115	EN16711-1	334385		----	
2121		----		----	
2129	In house	314239		----	
2135		291389		----	
2159		----		----	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	324530		----	
2311	EN16711-1	327908.5		----	
2314		----		----	
2330		----		----	
2347		----		----	
2350	EPA3052 / IEC62321-4,5	317600		----	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375	ISO16711-1	305433		----	
2379		----		----	
2380		----		----	
2385		----		----	
2406		----		----	
2464		----		----	
2476		----		----	
2492		----		----	
2509		----		----	
2553		----		----	
2564		----		----	
2590	EN16711-1	112285.10	G(0.01)	----	
2624		----		----	
2629	In house	376375		----	
2656	In house	detected		----	
2672	DIN13094-3	268100		----	
2678		----		----	
2703		----		----	
2743		----		----	
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146		----		----	
3160		----		----	
3183		----		----	
	normality	OK			
	n	13			
	outliers	1			
	mean (n)	328217.63			
	st.dev. (n)	32180.775	RSD = 10%		
	R(calc.)	90106.17			
	st.dev.(Horwitz)	(7762.771)			
	R(Horwitz)	(21735.76)			

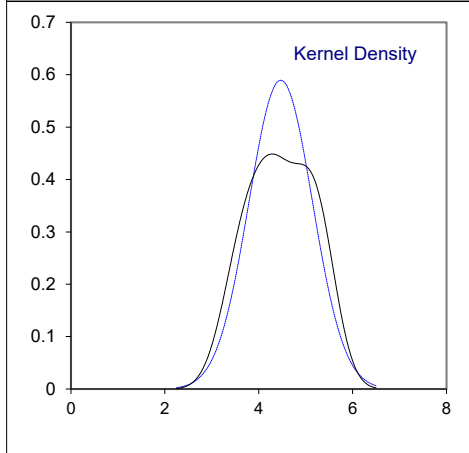
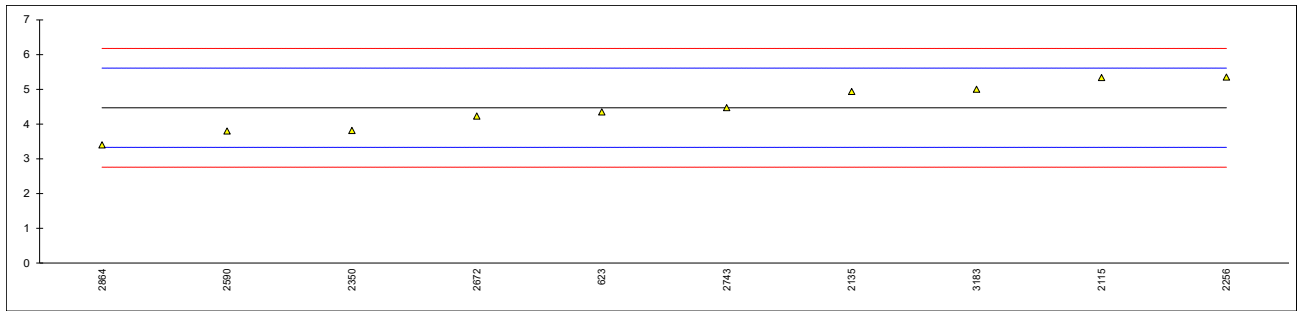


**APPENDIX 2 – CLASP (Lobster claw)**

Determination of Cadmium as Cd on sample #21630-2; results in mg/kg

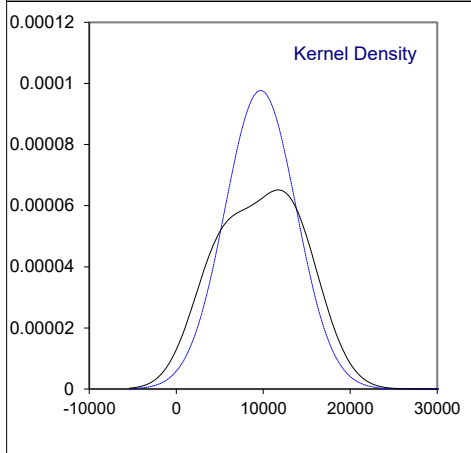
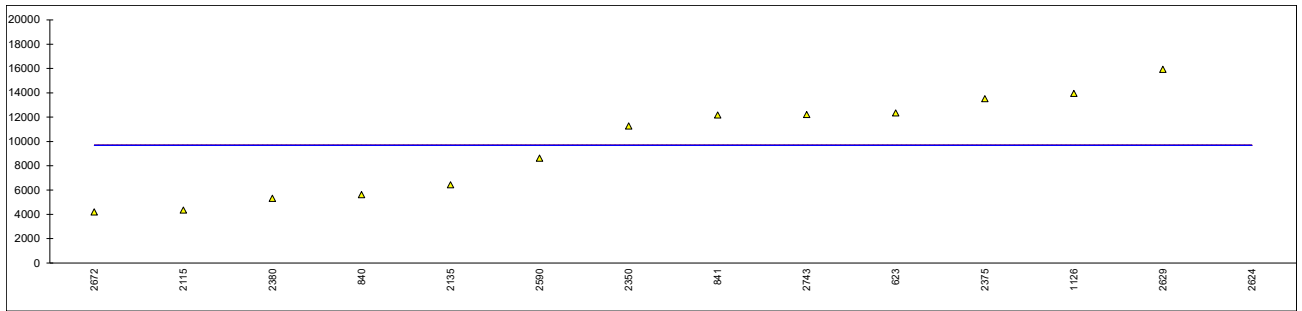
lab	method	value	mark	z(targ)	remarks
110	In house	not detected		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	4.35		-0.21	
840	In house	<5		----	
841	In house	<10		----	
1126		----		----	
1910	In house	not detected		----	
2115	EN16711-1	5.34		1.53	
2121		----		----	
2129		----		----	
2135		4.936		0.82	
2159	In house	<10		----	
2184	CPSC-CH-E1001-08.3	<10		----	
2216		----		----	
2256	CPSC-CH-E1001-08.3	5.35		1.54	
2266		----		----	
2310		----		----	
2311		----		----	
2314		----		----	
2330		----		----	
2347	In house	<5		----	
2350	EPA3052 / IEC62321-4,5	3.816		-1.14	
2357	In house	<5		----	
2363	C02.4	<5.0		----	
2365	In house	<5		----	
2366	C02.4.1	<5		----	
2369		<5		----	
2370		----		----	
2375	ISO16711-1	<10		----	
2379	IEC62321-4,5	not detected		----	
2380		----		----	
2385		----		----	
2406	ASTM F963-17	not detected		----	
2464		----		----	
2476		----		----	
2492		----		----	
2509	CPSC-CH-E1001-08	not detected		----	
2553		----		----	
2564		not detected		----	
2590	EN16711-1	3.80		-1.17	
2624		----		----	
2629	In house	<5		----	
2656	In house	not detected		----	
2672	DIN13094-3	4.230		-0.42	
2678		----		----	
2703		----		----	
2743		4.471234		0.00	
2789	CPSC-CH-E1001-08.3	not detected		----	
2810		----		----	
2829		not detected		----	
2851		not detected		----	
2864	In house	3.40		-1.87	
3146	ISO17294-2 / ISO17852-4	not detected		----	
3160	In house	not detected		----	
3183		5		0.93	
	normality	OK			
	n	10			
	outliers	0			
	mean (n)	4.4693			
	st.dev. (n)	0.67683	RSD = 15%		
	R(calc.)	1.8951			
	st.dev.(Horwitz)	0.57081			
	R(Horwitz)	1.5983			





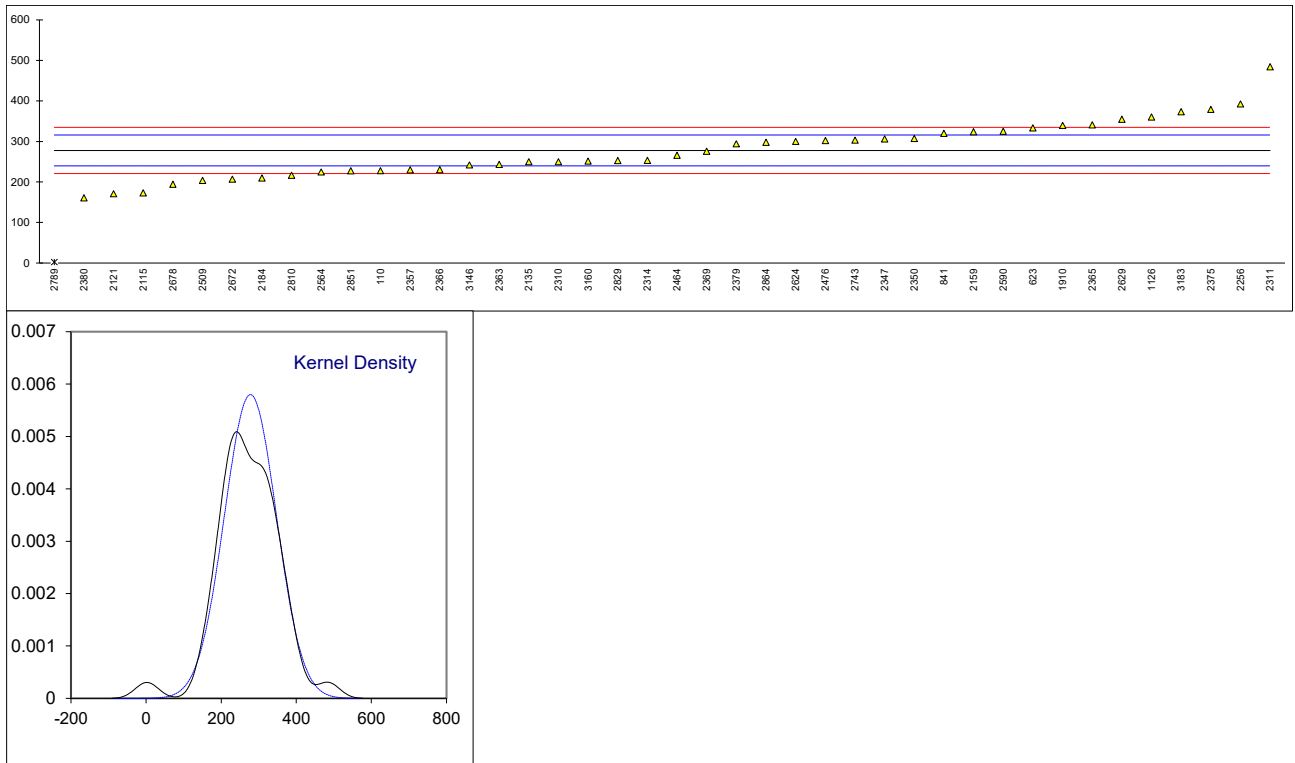
Determination of Copper as Cu on sample #21630-2; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	12347.24		----	
840	In house	5620		----	
841	In house	12175		----	
1126	In house	13950		----	
1910		----		----	
2115	EN16711-1	4347		----	
2121		----		----	
2129		----		----	
2135		6429.5		----	
2159		----		----	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310		----		----	
2311		----		----	
2314		----		----	
2330		----		----	
2347		----		----	
2350	EPA3052 / IEC62321-4,5	11270		----	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375	ISO16711-1	13520		----	
2379		----		----	
2380	CPSC-CH-E1001-08.3	5313.67		----	
2385		----		----	
2406		----		----	
2464		----		----	
2476		----		----	
2492		----		----	
2509		----		----	
2553		----		----	
2564		----		----	
2590	EN16711-1	8617.80		----	
2624	In house	166160	G(0.01)	----	
2629	In house	15935		----	
2656	In house	detected		----	
2672	DIN13094-3	4194		----	
2678		----		----	
2703		----		----	
2743		12207.2094	C	----	First reported 122.0721
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146		----		----	
3160		----		----	
3183		----		----	
	normality	OK			
	n	13			
	outliers	1			
	mean (n)	9686.647			
	st.dev. (n)	4083.8765	RSD = 42%		
	R(calc.)	11434.854			
	st.dev.(Horwitz)	(389.3271)			
	R(Horwitz)	(1090.116)			



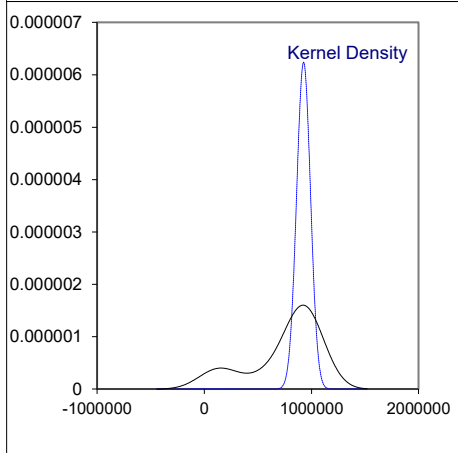
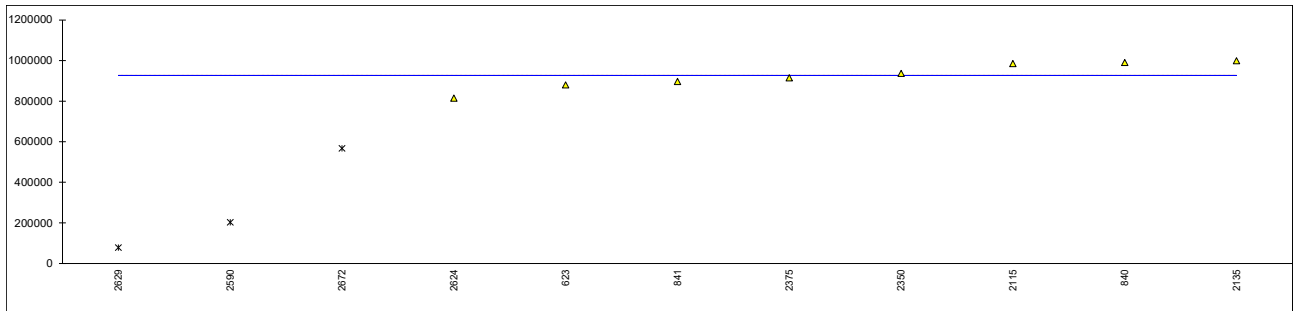
## Determination of Lead as Pb on sample #21630-2; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	227.7		-2.63	
210		----		----	
348		----		----	
551		----		----	
623	In house	333.11		2.90	
840		----		----	
841	In house	320		2.21	
1126	In house	360		4.31	
1910	In house	339.54		3.23	
2115	EN16711-1	173		-5.50	
2121		170.7		-5.62	
2129		----		----	
2135		249.73		-1.48	
2159	In house	324		2.42	
2184	CPSC-CH-E1001-08.3	209.7		-3.58	
2216		----		----	
2256	CPSC-CH-E1001-08.3	392.3		6.00	
2266		----		----	
2310	CPSC-CH-E1001-08.3	250		-1.46	
2311	EN16711-1	484		10.81	
2314	In house	253		-1.31	
2330		----		----	
2347	In house	306.2		1.49	
2350	EPA3052 / IEC62321-4,5	307.1		1.53	
2357	In house	229.8		-2.52	
2363	C02.4	243.4		-1.81	
2365	In house	340.7		3.30	
2366	C02.4.1	230.2		-2.50	
2369		275.6		-0.12	
2370	EPA3052 / IEC62321-4,5	not analyzed		----	
2375	ISO16711-1	379		5.31	
2379	IEC62321-4,5	294.07		0.85	
2380	CPSC-CH-E1001-08.3	160.58		-6.15	
2385		----		----	
2406	ASTM F963-17	not detected	C	----	First reported 110
2464	CPSC-CH-E1001-08.3	265.78		-0.63	
2476	CPSC-CH-E1001-08.3	302.2		1.28	
2492	In house	not analyzed		----	
2509	CPSC-CH-E1001-08	203.596204		-3.90	
2553		----		----	
2564		224.571		-2.80	
2590	EN16711-1	324.80		2.46	
2624	In house	300		1.16	
2629	In house	354.5		4.02	
2656	In house	detected		----	
2672	DIN13094-3	206.5		-3.74	
2678	CPSC-CH-E1001-08.3	194.44		-4.38	
2703		----		----	
2743		303.2711		1.33	
2789	CPSC-CH-E1001-08.3	2.09	R(0.05)	-14.47	
2810	CPSC-CH-E1001-08.3	216.292		-3.23	
2829		252.80		-1.32	
2851		227.2605		-2.66	
2864	In house	297.57		1.03	
3146	ISO17294-2 / ISO17852-4	241.7		-1.90	
3160	In house	251.39		-1.39	
3183		373		4.99	
	normality	OK			
	n	41			
	outliers	1			
	mean (n)	277.8805			
	st.dev. (n)	68.79990	RSD = 25%		
	R(calc.)	192.6397			
	st.dev.(Horwitz)	19.06078			
	R(Horwitz)	53.3702			



Determination of Zinc as Zn on sample #21630-2; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	879595.13		----	
840	In house	990069		----	
841	In house	896643		----	
1126		----		----	
1910		----		----	
2115	EN16711-1	984821		----	
2121		----		----	
2129		----		----	
2135		999000		----	
2159		----		----	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310		----		----	
2311		----		----	
2314		----		----	
2330		----		----	
2347		----		----	
2350	EPA3052 / IEC62321-4,5	936000		----	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375	ISO16711-1	914697		----	
2379		----		----	
2380		----		----	
2385		----		----	
2406		----		----	
2464		----		----	
2476		----		----	
2492		----		----	
2509		----		----	
2553		----		----	
2564		----		----	
2590	EN16711-1	203263.60	DG(0.01)	----	
2624	In house	814270		----	
2629	In house	78225	DG(0.01)	----	
2656	In house	detected		----	
2672	DIN13094-3	567000	G(0.05)	----	
2678		----		----	
2703		----		----	
2743		----		----	
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146		----		----	
3160		----		----	
3183		----		----	
	normality	OK			
	n	8			
	outliers	3			
	mean (n)	926886.9			
	st.dev. (n)	63935.29	RSD = 7%		
	R(calc.)	179018.8			
	st.dev.(Horwitz)	(18750.80)			
	R(Horwitz)	(52502.2)			

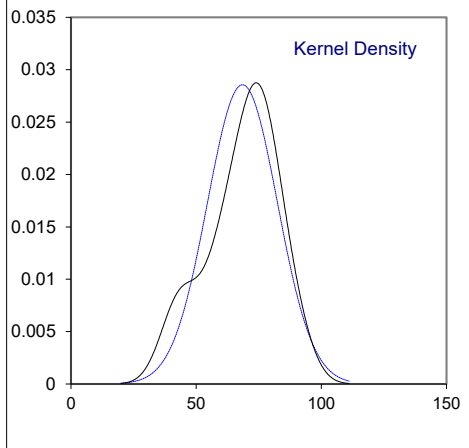
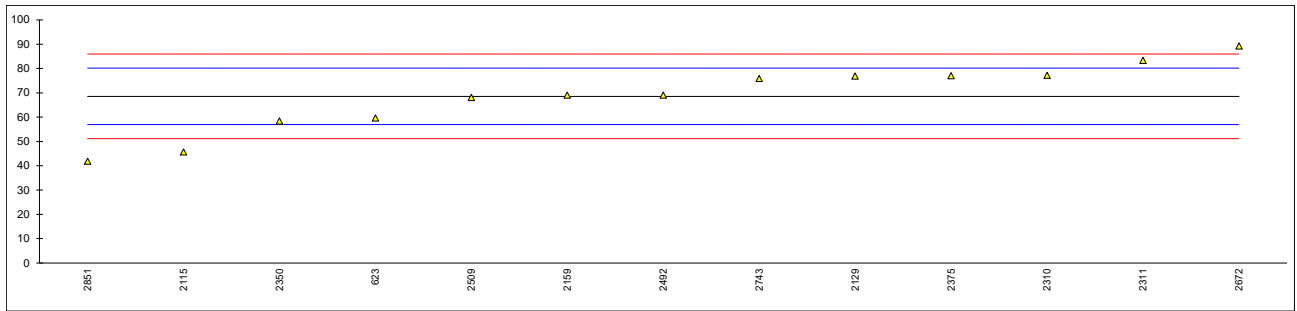


**APPENDIX 3 – LARGER RINGS (Extension rings)**

Determination of Arsenic as As on sample #21630-3; results in mg/kg

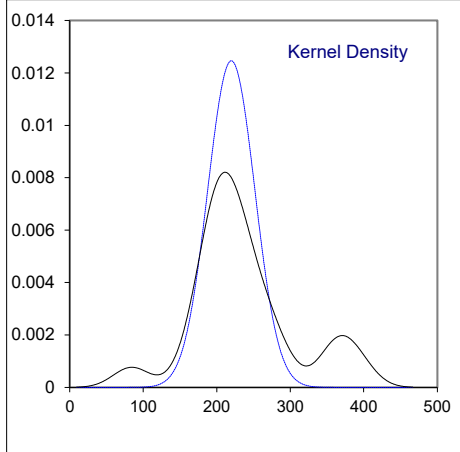
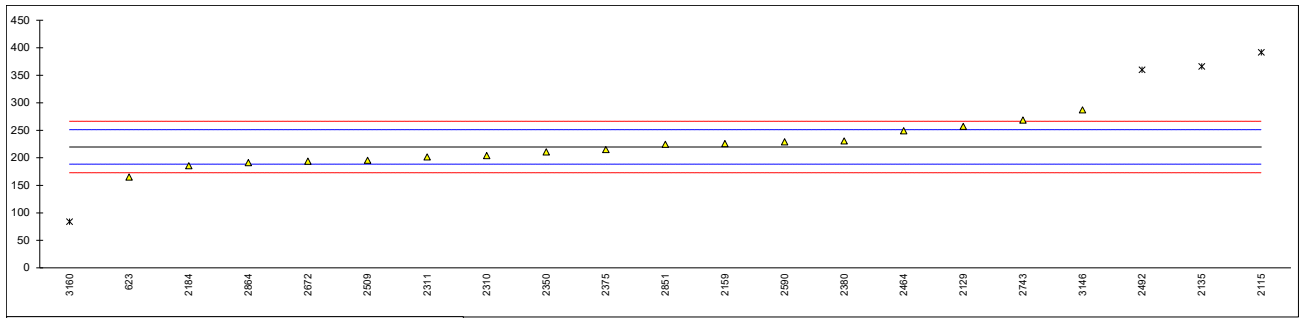
lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	59.67	C	-1.53	First reported "not detected"
840		----		----	
841		----		----	
1126		----		----	
1910		----		----	
2115	EN16711-1	45.61		-3.95	
2121		----		----	
2129	In house	76.85		1.43	
2135		----		----	
2159	In house	69		0.08	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	77.2		1.49	
2311	EN16711-1	83.36		2.55	
2314		----		----	
2330		----		----	
2347		----		----	
2350	EPA3052 / IEC62321-4,5	58.38		-1.75	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375	ISO16711-1	77.0		1.46	
2379		----		----	
2380		----		----	
2385		----		----	
2406	ASTM F963-17	not detected		----	
2464		----		----	
2476		----		----	
2492	In house	69.04		0.08	
2509	CPSC-CH-E1001-08	68.083895		-0.08	
2553		----		----	
2564		----		----	
2590		----		----	
2624		----		----	
2629		----		----	
2656	In house	detected		----	
2672	DIN13094-3	89.30		3.57	
2678		----		----	
2703		----		----	
2743		75.8474726		1.26	
2789		----		----	
2810		----		----	
2829		----		----	
2851		41.8098		-4.61	
2864		----		----	
3146		----		----	
3160		----		----	
3183		----		----	
	normality	OK			
	n	13			
	outliers	0			
	mean (n)	68.5501			
	st.dev. (n)	13.97167	RSD = 20%		
	R(calc.)	39.1207			
	st.dev.(Horwitz)	5.80472			
	R(Horwitz)	16.2532			





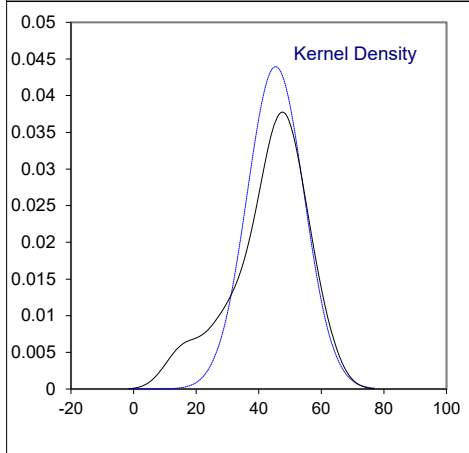
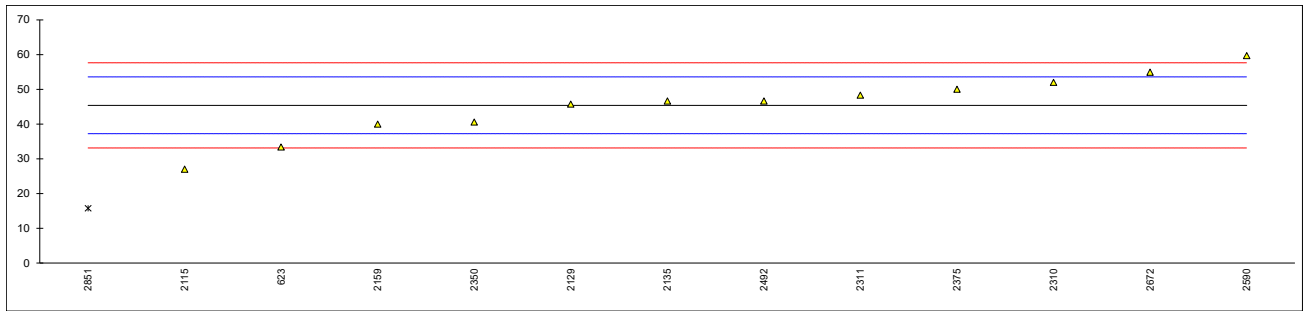
## Determination of Chromium as Cr on sample #21630-3; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	164.99		-3.51	
840		----		----	
841		----		----	
1126		----		----	
1910		----		----	
2115	EN16711-1	391.9	C	11.02	First reported 405.2
2121		----		----	
2129	In house	257.2		2.40	
2135		366.2		9.38	
2159	In house	226		0.40	
2184	CPSC-CH-E1001-08.3	185.8		-2.17	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	204		-1.01	
2311	EN16711-1	201.70		-1.16	
2314		----		----	
2330		----		----	
2347		----		----	
2350	EPA3052 / IEC62321-4,5	210.8		-0.57	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375	ISO16711-1	215.0		-0.30	
2379		----		----	
2380	CPSC-CH-E1001-08.3	230.66		0.70	
2385		----		----	
2406	ASTM F963-17	not detected	C	----	First reported 520
2464	CPSC-CH-E1001-08.3	249.28		1.89	
2476		----		----	
2492	In house	359.87		8.97	
2509	CPSC-CH-E1001-08	195.42544		-1.56	
2553		----		----	
2564		----		----	
2590	EN16711-1	229.30		0.61	
2624		----		----	
2629		----		----	
2656	In house	not detected		----	
2672	DIN13094-3	194.0		-1.65	
2678		----		----	
2703		----		----	
2743		268.648833		3.13	
2789		----		----	
2810		----		----	
2829		----		----	
2851		224.5706		0.31	
2864	In house	191.31		-1.82	
3146	ISO17294-2 / ISO17852-4	287.2		4.32	
3160	In house	84.00		-8.69	
3183		----		----	
	normality	OK			
	n	17			
	outliers	4			
	mean (n)	219.7579			
	st.dev. (n)	31.99514	RSD = 15%		
	R(calc.)	89.5864			
	st.dev.(Horwitz)	15.61589			
	R(Horwitz)	43.7245			



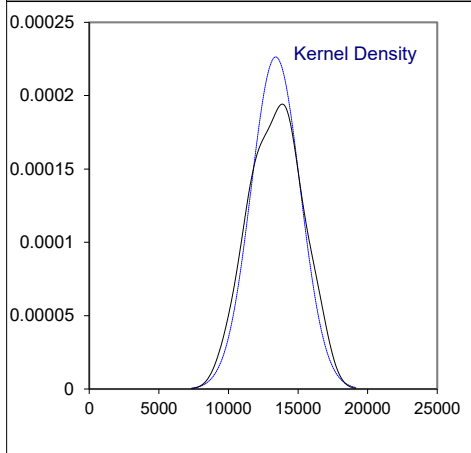
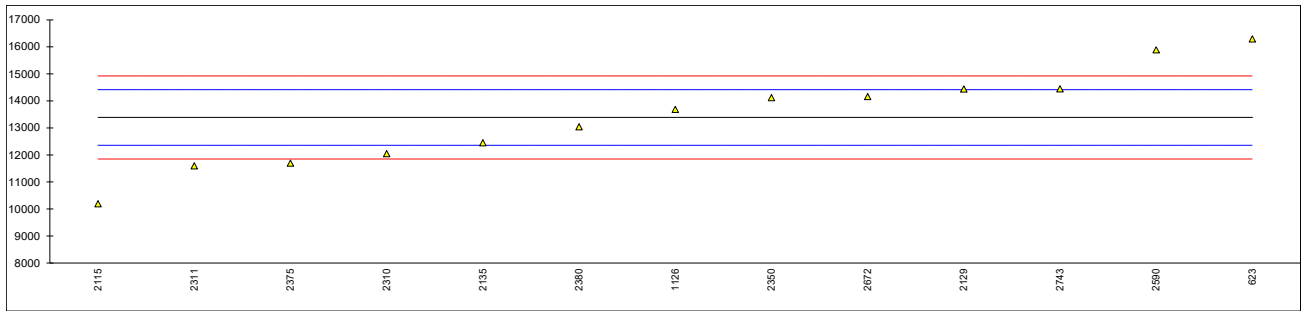
Determination of Cobalt as Co on sample #21630-3; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	33.39		-2.94	
840		----		----	
841		----		----	
1126		----		----	
1910		----		----	
2115	EN16711-1	27		-4.50	
2121		----		----	
2129	In house	45.75		0.08	
2135		46.605		0.29	
2159	In house	40		-1.32	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	52		1.61	
2311	EN16711-1	48.33		0.71	
2314		----		----	
2330		----		----	
2347		----		----	
2350	EPA3052 / IEC62321-4,5	40.59		-1.18	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375	ISO16711-1	50.0		1.12	
2379		----		----	
2380		----		----	
2385		----		----	
2406		----		----	
2464		----	W	----	Test result withdrawn, reported 80.13
2476		----		----	
2492	In house	46.61		0.29	
2509		----		----	
2553		----		----	
2564		----		----	
2590	EN16711-1	59.70		3.49	
2624		----		----	
2629		----		----	
2656	In house	not detected		----	
2672	DIN13094-3	54.90		2.32	
2678		----		----	
2703		----		----	
2743		----		----	
2789		----		----	
2810		----		----	
2829		----		----	
2851		15.7362	G(0.05)	-7.25	
2864		----		----	
3146		----		----	
3160		----		----	
3183		----		----	
	normality	OK			
	n	12			
	outliers	1			
	mean (n)	45.4063			
	st.dev. (n)	9.07634	RSD = 20%		
	R(calc.)	25.4138			
	st.dev.(Horwitz)	4.09086			
	R(Horwitz)	11.4544			



Determination of Copper as Cu on sample #21630-3; results in mg/kg

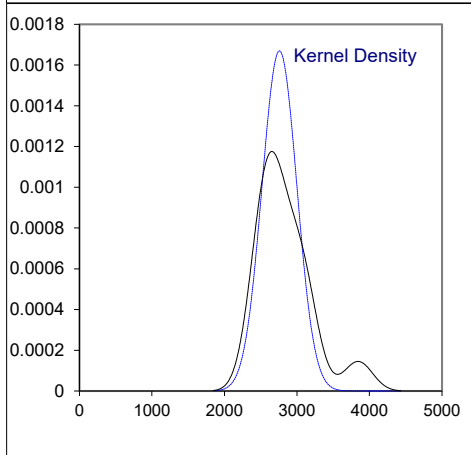
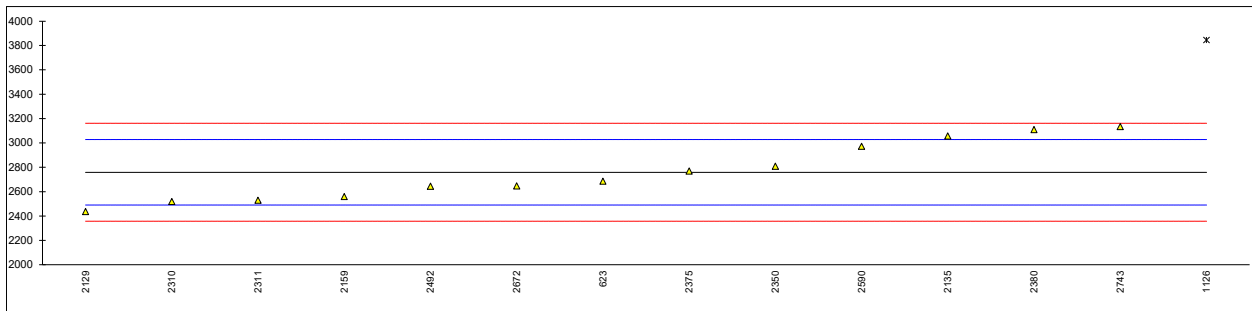
lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	16296.25		5.67	
840		----		----	
841		----		----	
1126	In house	13685		0.58	
1910		----		----	
2115	EN16711-1	10191		-6.24	
2121		----		----	
2129	In house	14443		2.06	
2135		12451		-1.83	
2159		----		----	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	12048		-2.62	
2311	EN16711-1	11597.41		-3.50	
2314		----		----	
2330		----		----	
2347		----		----	
2350	EPA3052 / IEC62321-4,5	14120		1.43	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375	ISO16711-1	11693		-3.31	
2379		----		----	
2380	CPSC-CH-E1001-08.3	13043.23		-0.68	
2385		----		----	
2406		----		----	
2464		----		----	
2476		----		----	
2492		----		----	
2509		----		----	
2553		----		----	
2564		----		----	
2590	EN16711-1	15889.70		4.88	
2624		----		----	
2629		----		----	
2656	In house	detected		----	
2672	DIN13094-3	14160		1.50	
2678		----		----	
2703		----		----	
2743		14445.0809	C	2.06	First reported 144.450809
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146		----		----	
3160		----		----	
3183		----		----	
	normality	OK			
	n	13			
	outliers	0			
	mean (n)	13389.44			
	st.dev. (n)	1762.085	RSD = 13%		
	R(calc.)	4933.85			
	st.dev.(Horwitz)	512.558			
	R(Horwitz)	1435.16			



Determination of Manganese as Mn on sample #21630-3; results in mg/kg

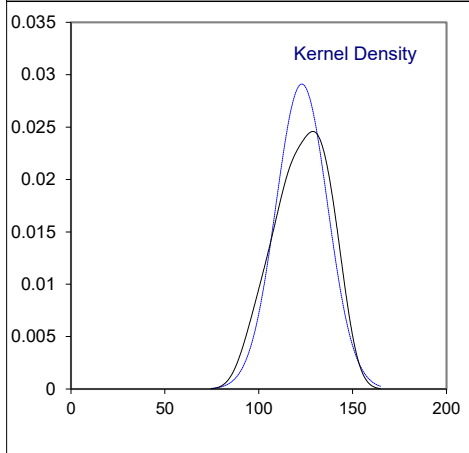
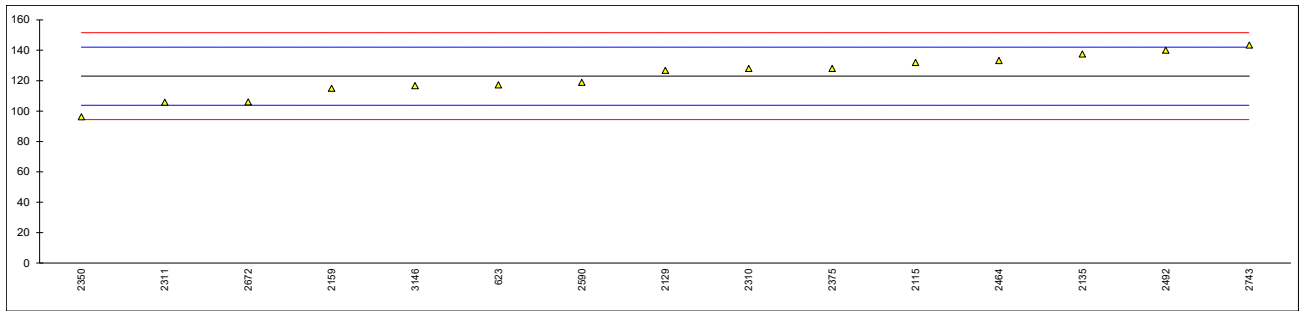
lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	2685.87		-0.55	
840		----		----	
841		----		----	
1126	In house	3845	G(0.05)	8.10	
1910		----		----	
2115		----		----	
2121		----		----	
2129	In house	2436		-2.41	
2135		3056		2.22	
2159	In house	2559		-1.49	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	2519		-1.79	
2311	EN16711-1	2529.23		-1.72	
2314		----		----	
2330		----		----	
2347		----		----	
2350	EPA3052 / IEC62321-4,5	2809		0.37	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375	ISO16711-1	2769.0		0.07	
2379		----		----	
2380	CPSC-CH-E1001-08.3	3110.19		2.62	
2385		----		----	
2406		----		----	
2464		----	W	----	Test result withdrawn, reported 3838.02
2476		----		----	
2492	In house	2644.78		-0.85	
2509		----		----	
2553		----		----	
2564		----		----	
2590	EN16711-1	2971.50		1.58	
2624		----		----	
2629		----		----	
2656	In house	detected		----	
2672	DIN13094-3	2646		-0.84	
2678		----		----	
2703		----		----	
2743		3133.97099	C	2.80	First reported 313.397099
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146		----		----	
3160		----		----	
3183		----		----	
	normality	OK			
	n	13			
	outliers	1			
	mean (n)	2759.196			
	st.dev. (n)	239.0587	RSD = 9%		
	R(calc.)	669.364			
	st.dev.(Horwitz)	133.9717			
	R(Horwitz)	375.121			





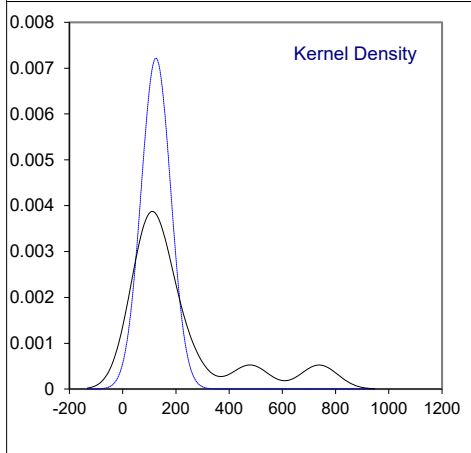
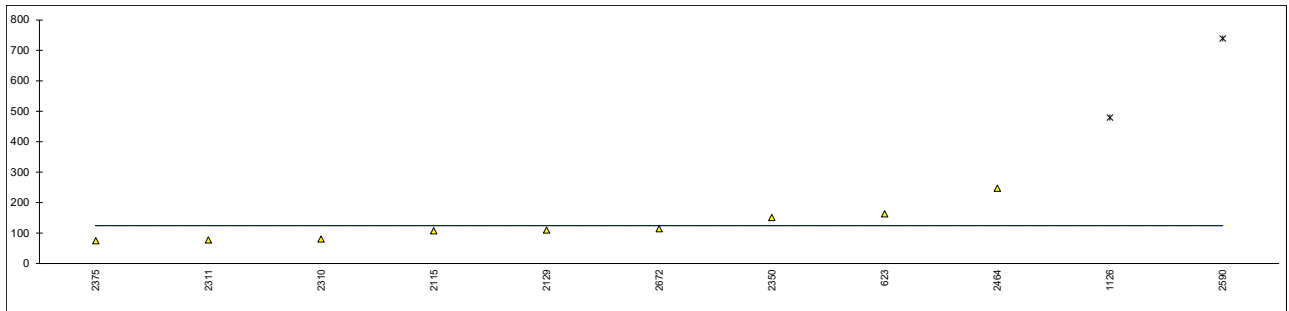
## Determination of Nickel as Ni on sample #21630-3; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	117.17	C	-0.61	First reported 92.12
840		----		----	
841		----		----	
1126		----		----	
1910		----		----	
2115	EN16711-1	131.99	C	0.95	First reported 67.56
2121		----		----	
2129	In house	126.77		0.40	
2135		137.55		1.53	
2159	In house	115		-0.84	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	128		0.53	
2311	EN16711-1	105.76		-1.81	
2314		----		----	
2330		----		----	
2347		----		----	
2350	EPA3052 / IEC62321-4,5	96.2		-2.81	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375	ISO16711-1	128.0		0.53	
2379		----		----	
2380		----		----	
2385		----		----	
2406		----		----	
2464	CPSC-CH-E1001-08.3	133.30		1.08	
2476		----		----	
2492	In house	140.03		1.79	
2509		----		----	
2553		----		----	
2564		----		----	
2590	EN16711-1	118.80		-0.44	
2624		----		----	
2629		----		----	
2656	In house	not detected		----	
2672	DIN13094-3	106.0		-1.78	
2678		----		----	
2703		----		----	
2743		143.372924		2.14	
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146	ISO17294-2 / ISO17852-4	116.7		-0.66	
3160		----		----	
3183		----		----	
	normality	OK			
	n	15			
	outliers	0			
	mean (n)	122.9762			
	st.dev. (n)	13.70003	RSD = 11%		
	R(calc.)	38.3601			
	st.dev.(Horwitz)	9.53656			
	R(Horwitz)	26.7024			



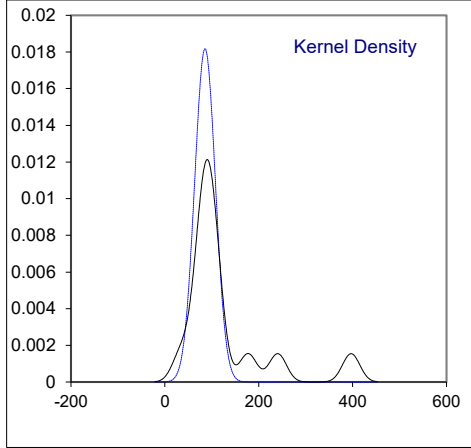
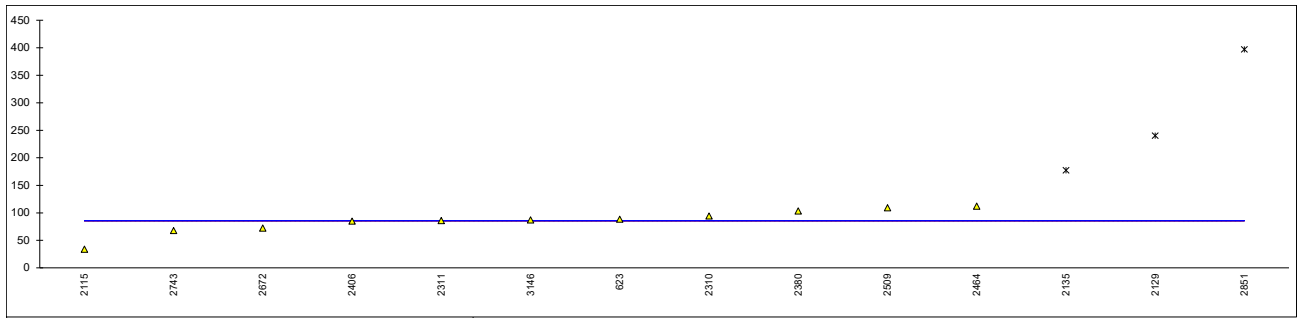
Determination of Zinc as Zn on sample #21630-3; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	162.70		----	
840		----		----	
841		----		----	
1126	In house	479	G(0.01)	----	
1910		----		----	
2115	EN16711-1	107.47		----	
2121		----		----	
2129	In house	109.75		----	
2135		----		----	
2159		----		----	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	80		----	
2311	EN16711-1	77.07		----	
2314		----		----	
2330		----		----	
2347		----		----	
2350	EPA3052 / IEC62321-4,5	150.9		----	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375	ISO16711-1	75.0		----	
2379		----		----	
2380		----		----	
2385		----		----	
2406		----		----	
2464	CPSC-CH-E1001-08.3	246.95		----	
2476		----		----	
2492		----		----	
2509		----		----	
2553		----		----	
2564		----		----	
2590	EN16711-1	738.80	G(0.01)	----	
2624		----		----	
2629		----		----	
2656	In house	not detected		----	
2672	DIN13094-3	114.0		----	
2678		----		----	
2703		----		----	
2743		----		----	
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146		----		----	
3160		----		----	
3183		----		----	
	normality	not OK			
	n	9			
	outliers	2			
	mean (n)	124.8711			
	st.dev. (n)	55.27986	RSD = 44%		
	R(calc.)	154.7836			
	st.dev.(Horwitz)	(9.66124)			
	R(Horwitz)	(27.0515)			



**APPENDIX 4 – LARGE CONNECTION RINGS (at the end of the ball chain)**  
 Determination of Chromium as Cr on sample #21630-4; results in mg/kg

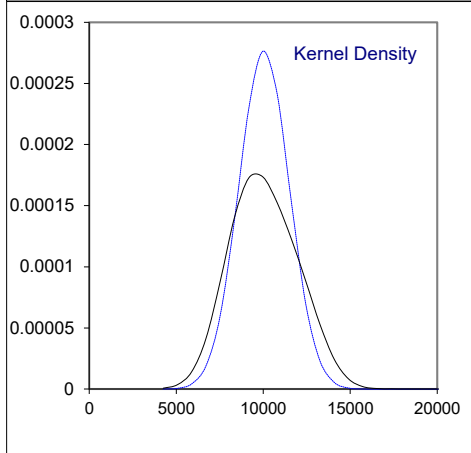
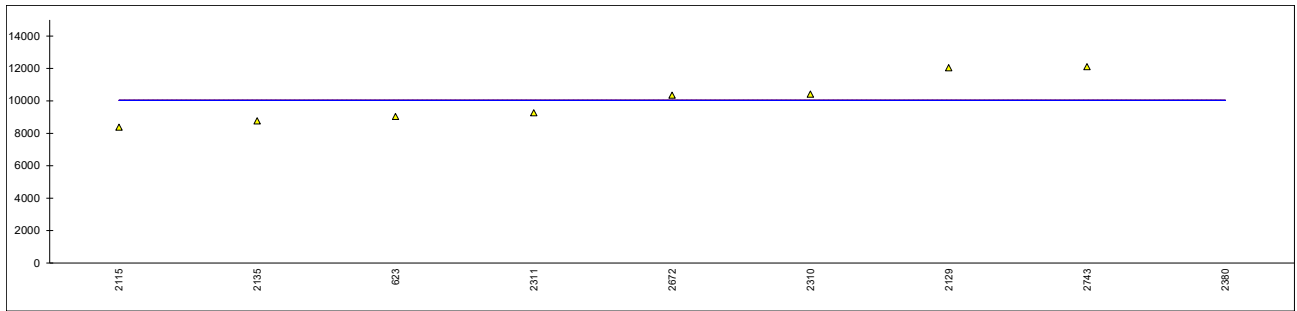
lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	88.50		----	
840		----		----	
841		----		----	
1126		----		----	
1910		----		----	
2115	EN16711-1	34.1		----	
2121		----		----	
2129	In house	240.5	G(0.05)	----	
2135		177.3	G(0.05)	----	
2159		----		----	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	94.3		----	
2311	EN16711-1	86.20		----	
2314		----		----	
2330		----		----	
2347		----		----	
2350		----		----	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375		----		----	
2379		----		----	
2380	CPSC-CH-E1001-08.3	103.31		----	
2385		----		----	
2406	ASTM F963-17	85		----	
2464	CPSC-CH-E1001-08.3	112.22		----	
2476		----		----	
2492		----		----	
2509	CPSC-CH-E1001-08	109.424788		----	
2553		----		----	
2564		----		----	
2590		----		----	
2624		----		----	
2629		----		----	
2656	In house	not detected		----	
2672	DIN13094-3	72.35		----	
2678		----		----	
2703		----		----	
2743		67.9558824		----	
2789		----		----	
2810		----		----	
2829		----		----	
2851		397.0212	G(0.01)	----	
2864		----		----	
3146	ISO17294-2 / ISO17852-4	87.24		----	
3160		----		----	
3183		----		----	
	normality	not OK			
	n	11			
	outliers	3			
	mean (n)	85.5092			
	st.dev. (n)	21.94638	RSD = 26%		
	R(calc.)	61.4499			
	st.dev.(Horwitz)	(7.00383)			
	R(Horwitz)	(19.6107)			



Determination of Copper as Cu on sample #21630-4; results in mg/kg

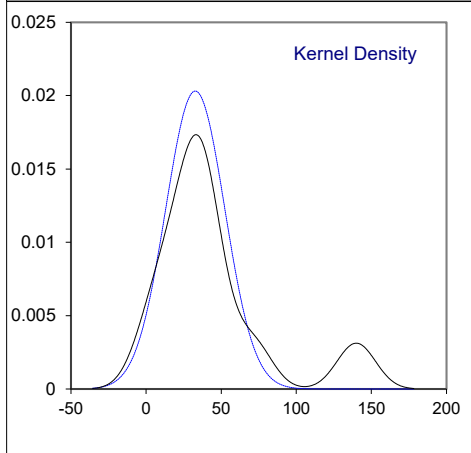
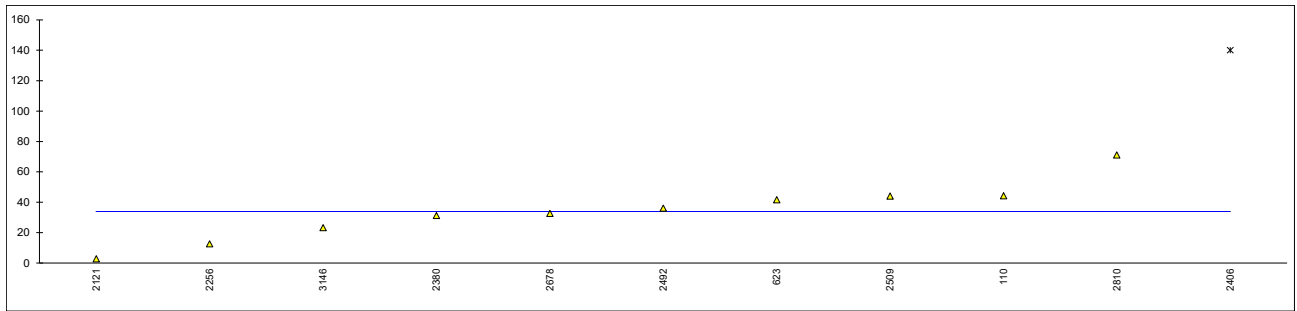
lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	9040.49		----	
840		----		----	
841		----		----	
1126		----		----	
1910		----		----	
2115	EN16711-1	8379		----	
2121		----		----	
2129	In house	12049		----	
2135		8762		----	
2159		----		----	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	10415		----	
2311	EN16711-1	9264.09		----	
2314		----		----	
2330		----		----	
2347		----		----	
2350		----		----	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375		----		----	
2379		----		----	
2380	CPSC-CH-E1001-08.3	209118	G(0.01)	----	
2385		----		----	
2406		----		----	
2464		----		----	
2476		----		----	
2492		----		----	
2509		----		----	
2553		----		----	
2564		----		----	
2590		----		----	
2624		----		----	
2629		----		----	
2656	In house	detected		----	
2672	DIN13094-3	10350		----	
2678		----		----	
2703		----		----	
2743		12114.3908	C	----	First reported 121.143908
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146		----		----	
3160		----		----	
3183		----		----	
	normality	OK			
	n	8			
	outliers	1			
	mean (n)	10046.75			
	st.dev. (n)	1442.656	RSD = 14%		
	R(calc.)	4039.44			
	st.dev.(Horwitz)	(401.588)			
	R(Horwitz)	(1124.45)			





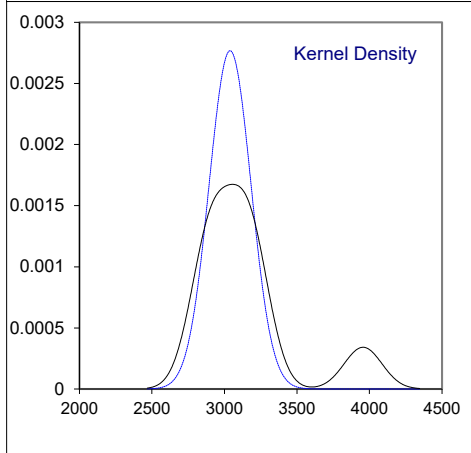
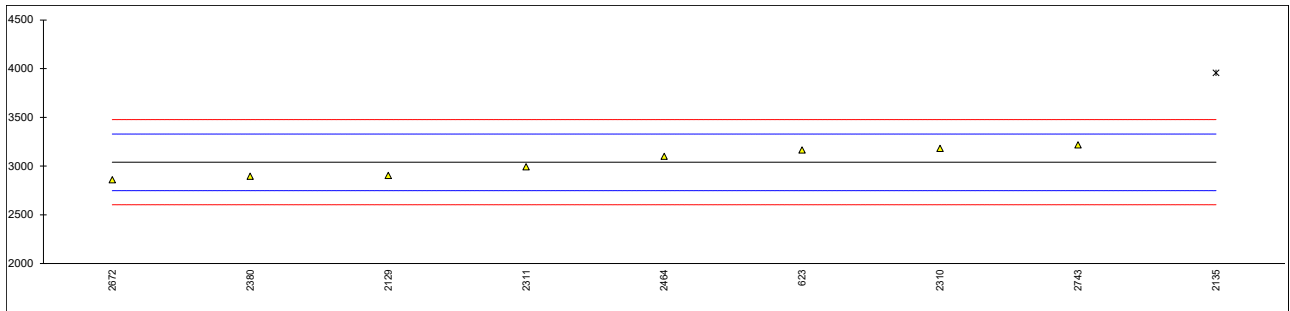
## Determination of Lead as Pb on sample #21630-4; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	44.2		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	41.59		----	
840		----		----	
841		----		----	
1126		----		----	
1910	In house	not detected		----	
2115		----		----	
2121		2.69		----	
2129	In house	<5		----	
2135		----		----	
2159		----		----	
2184		----		----	
2216		----		----	
2256	CPSC-CH-E1001-08.3	12.6		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	not detected		----	
2311	EN16711-1	<10		----	
2314	In house	not detected		----	
2330		----		----	
2347		----		----	
2350		----		----	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375		----		----	
2379	IEC62321-4,5	not detected		----	
2380	CPSC-CH-E1001-08.3	31.20		----	
2385		----		----	
2406	ASTM F963-17	140	G(0.01)	----	
2464		----		----	
2476		----		----	
2492	In house	36.03		----	
2509	CPSC-CH-E1001-08	43.9486829		----	
2553		----		----	
2564		not detected		----	
2590		----		----	
2624		----		----	
2629		----		----	
2656	In house	detected		----	
2672	DIN13094-3	not quantified		----	
2678	CPSC-CH-E1001-08.3	32.57		----	
2703		----		----	
2743		----		----	
2789		----		----	
2810	CPSC-CH-E1001-08.3	71.049		----	
2829		----		----	
2851		not detected		----	
2864		----		----	
3146	ISO17294-2 / ISO17852-4	23.24		----	
3160		----		----	
3183		----		----	
	normality	suspect			
	n	10			
	outliers	1			
	mean (n)	33.9118			
	st.dev. (n)	18.85982	RSD = 56%		
	R(calc.)	52.8075			
	st.dev.(Horwitz)	(3.19249)			
	R(Horwitz)	(8.9390)			



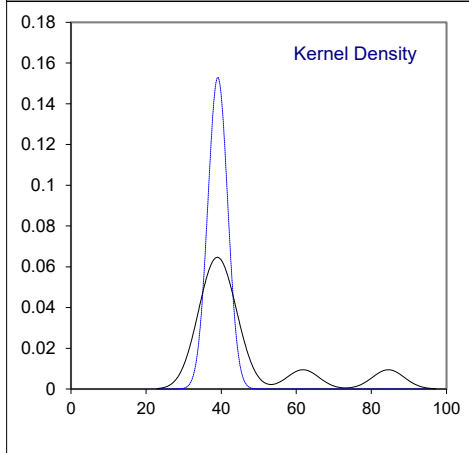
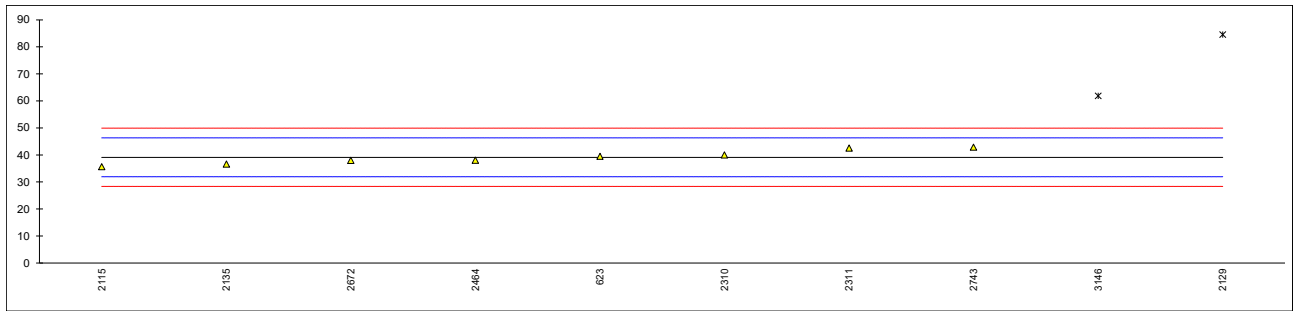
Determination of Manganese as Mn on sample #21630-4; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	3165.309		0.87	
840		----		----	
841		----		----	
1126		----		----	
1910		----		----	
2115		----		----	
2121		----		----	
2129	In house	2904		-0.93	
2135		3956	G(0.01)	6.30	
2159		----		----	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	3181		0.97	
2311	EN16711-1	2992.39		-0.32	
2314		----		----	
2330		----		----	
2347		----		----	
2350		----		----	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375		----		----	
2379		----		----	
2380	CPSC-CH-E1001-08.3	2895.19		-0.99	
2385		----		----	
2406		----		----	
2464	CPSC-CH-E1001-08.3	3099.40		0.41	
2476		----		----	
2492		----		----	
2509		----		----	
2553		----		----	
2564		----		----	
2590		----		----	
2624		----		----	
2629		----		----	
2656	In house	detected		----	
2672	DIN13094-3	2859		-1.24	
2678		----		----	
2703		----		----	
2743		3218.47689	C	1.23	First reported 321.847689
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146		----		----	
3160		----		----	
3183		----		----	
	normality	OK			
	n	8			
	outliers	1			
	mean (n)	3039.345			
	st.dev. (n)	144.1507	RSD = 5%		
	R(calc.)	403.622			
	st.dev.(Horwitz)	145.4419			
	R(Horwitz)	407.237			



Determination of Nickel as Ni on sample #21630-4; results in mg/kg

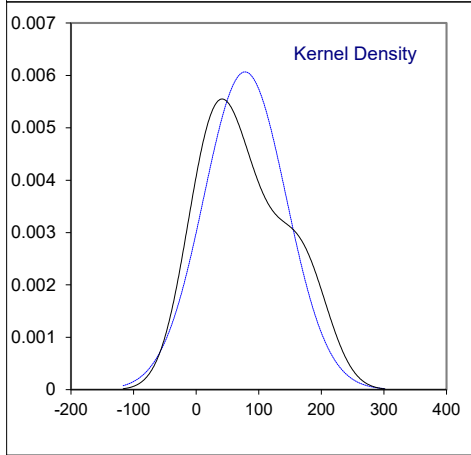
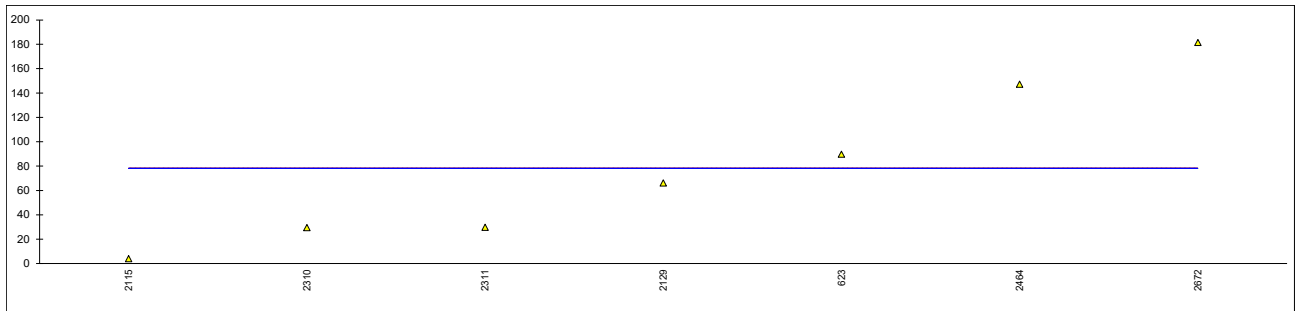
lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	39.51		0.11	
840		----		----	
841		----		----	
1126		----		----	
1910		----		----	
2115	EN16711-1	35.6	C	-0.98	First reported 15
2121		----		----	
2129	In house	84.55	G(0.01)	12.60	
2135		36.59		-0.70	
2159		----		----	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	40		0.24	
2311	EN16711-1	42.51		0.94	
2314		----		----	
2330		----		----	
2347		----		----	
2350		----		----	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375		----		----	
2379		----		----	
2380		----		----	
2385		----		----	
2406		----		----	
2464	CPSC-CH-E1001-08.3	38.04		-0.30	
2476		----		----	
2492		----		----	
2509		----		----	
2553		----		----	
2564		----		----	
2590		----		----	
2624		----		----	
2629		----		----	
2656	In house	not detected		----	
2672	DIN13094-3	37.95		-0.33	
2678		----		----	
2703		----		----	
2743		42.8434874		1.03	
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146	ISO17294-2 / ISO17852-4	61.83	G(0.01)	6.30	
3160		----		----	
3183		----		----	
	normality	OK			
	n	8			
	outliers	2			
	mean (n)	39.1304			
	st.dev. (n)	2.60792	RSD = 7%		
	R(calc.)	7.3022			
	st.dev.(Horwitz)	3.60527			
	R(Horwitz)	10.0947			



Determination of Zinc as Zn on sample #21630-4; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		----		----	
210		----		----	
348		----		----	
551		----		----	
623	In house	89.73		----	
840		----		----	
841		----		----	
1126		----		----	
1910		----		----	
2115	EN16711-1	3.94		----	
2121		----		----	
2129	In house	66.15		----	
2135		----		----	
2159		----		----	
2184		----		----	
2216		----		----	
2256		----		----	
2266		----		----	
2310	CPSC-CH-E1001-08.3	29.5		----	
2311	EN16711-1	29.70		----	
2314		----		----	
2330		----		----	
2347		----		----	
2350		----		----	
2357		----		----	
2363		----		----	
2365		----		----	
2366		----		----	
2369		----		----	
2370		----		----	
2375		----		----	
2379		----		----	
2380		----		----	
2385		----		----	
2406		----		----	
2464	CPSC-CH-E1001-08.3	147.26		----	
2476		----		----	
2492		----		----	
2509		----		----	
2553		----		----	
2564		----		----	
2590		----		----	
2624		----		----	
2629		----		----	
2656	In house	not detected		----	
2672	DIN13094-3	181.5		----	
2678		----		----	
2703		----		----	
2743		----		----	
2789		----		----	
2810		----		----	
2829		----		----	
2851		----		----	
2864		----		----	
3146		----		----	
3160		----		----	
3183		----		----	
	normality	unknown			
	n	7			
	outliers	0			
	mean (n)	78.2543			
	st.dev. (n)	65.77333	RSD = 84%		
	R(calc.)	184.1653			
	st.dev.(Horwitz)	(6.49571)			
	R(Horwitz)	(18.1880)			





**APPENDIX 5**

**Reported Total Metals (Not identified parts of the necklace) in sample #21630; results in mg/kg**

lab	Sb	As	Cd	Cr	Co	Cu	Pb	Mn	Hg	Ni	Se	Sr	Zn
110	----	----	----	----	----	----	----	----	----	----	----	----	----
210	----	----	----	----	----	----	----	----	----	----	----	----	----
348	----	----	----	----	----	----	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----	----	----	----	----	----	----
623	----	----	----	----	----	----	----	----	----	----	----	----	----
840	----	----	----	----	----	----	----	----	----	----	----	----	----
841	----	----	----	----	----	----	----	----	----	----	----	----	----
1126	----	----	----	----	----	----	----	----	----	----	----	----	----
1910	----	----	----	----	----	----	----	----	----	----	----	----	----
2115	----	----	----	----	----	----	----	----	----	----	----	----	----
2121	----	----	----	----	----	----	----	----	----	----	----	----	----
2129	----	----	----	----	----	----	----	----	----	----	----	----	----
2135	----	----	----	----	----	----	----	----	----	----	----	----	----
2159	----	----	----	----	----	----	----	----	----	----	----	----	----
2184	----	----	----	----	----	----	----	----	----	----	----	----	----
2216	not det.	13	7	420	----	----	78	----	not det.	131	not det.	----	----
2256	----	----	----	----	----	----	----	----	----	----	----	----	----
2266	not det.	24.1	not det.	575.4	15.3	346683.1	77.3	1150.8	not det.	87.0	not det.	not det.	380170.5
2310	----	----	----	----	----	----	----	----	----	----	----	----	----
2311	----	----	----	----	----	----	----	----	----	----	----	----	----
2314	----	----	----	----	----	----	----	----	----	----	----	----	----
2330	----	----	----	----	----	----	42.12	----	----	----	----	----	----
2347	----	----	----	----	----	----	----	----	----	----	----	----	----
2350	----	----	----	----	----	----	----	----	----	----	----	----	----
2357	----	----	----	----	----	----	----	----	----	----	----	----	----
2363	----	----	----	----	----	----	----	----	----	----	----	----	----
2365	----	----	----	----	----	----	----	----	----	----	----	----	----
2366	----	----	----	----	----	----	----	----	----	----	----	----	----
2369	----	----	----	----	----	----	----	----	----	----	----	----	----
2370	----	----	----	----	----	----	----	----	----	----	----	----	----
2375	----	----	----	----	----	----	----	----	----	----	----	----	----
2379	----	----	----	----	----	----	----	----	----	----	----	----	----
2380	----	----	----	----	----	----	----	----	----	----	----	----	----
2385	<10	<10	5.90	403	10.4	406000	73.6	1035	<0.5	54.8	<10	<20	336000
2406	----	----	----	----	----	----	----	----	----	----	----	----	----
2464	----	----	----	----	----	----	----	----	----	----	----	----	----
2476	----	----	----	----	----	----	----	----	----	----	----	----	----
2492	----	----	----	----	----	----	----	----	----	----	----	----	----
2509	----	----	----	----	----	----	----	----	----	----	----	----	----
2553	0.36	2.58	4.25	4375	20.5	1387.5	557.5	5375	0.1	311.25	0	0.25	937500
2564	----	----	----	----	----	----	----	----	----	----	----	----	----
2590	----	----	----	----	----	----	----	----	----	----	----	----	----
2624	----	----	----	----	----	----	----	----	----	----	----	----	----
2629	----	----	----	----	----	----	----	----	----	----	----	----	----
2656	----	----	----	----	----	----	----	----	----	----	----	----	----
2672	----	----	----	----	----	----	----	----	----	----	----	----	----
2678	----	----	----	----	----	----	----	----	----	----	----	----	----
2703	----	0.0	27.2	72.5	21.0	----	8.1	----	----	----	----	6772.8	306516.3
2743	----	----	----	----	----	----	----	----	----	----	----	----	----
2789	----	----	----	----	----	----	----	----	----	----	----	----	----
2810	----	----	----	----	----	----	----	----	----	----	----	----	----
2829	----	----	----	----	----	----	----	----	----	----	----	----	----
2851	----	----	----	----	----	----	----	----	----	----	----	----	----
2864	----	----	----	----	----	----	----	----	----	----	----	----	----
3146	----	----	----	----	----	----	----	----	----	----	----	----	----
3160	----	----	----	----	----	----	----	----	----	----	----	----	----
3183	----	----	----	----	----	----	----	----	----	----	----	----	----

**APPENDIX 6****Other reported Metals in sample #21630-1; results in mg/kg**

lab	Sb	As	Cr	Co	Mn	Hg	Se	Sr
110	----	----	----	----	----	----	----	----
210	----	----	----	----	----	----	----	----
348	----	----	<5	----	----	----	----	----
551	----	----	----	----	----	----	----	----
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected	----
840	<10	<10	<10	<10	<10	<5	<10	<10
841	----	----	----	----	----	----	----	----
1126	----	----	----	----	----	----	----	----
1910	----	----	----	----	----	----	----	----
2115	----	----	----	----	----	----	----	----
2121	----	----	----	----	----	----	----	----
2129	<5	<5	<5	<5	<5	<5	<50	<50
2135	----	----	----	1.529	----	----	----	----
2159	<10	<5	<10	<10	<10	<5	<10	<10
2184	----	----	24.4	----	----	<10	----	----
2216	----	----	----	----	----	----	----	----
2256	----	----	----	----	----	----	----	----
2266	----	----	----	----	----	----	----	----
2310	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2311	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2314	----	----	----	----	----	----	----	----
2330	----	----	----	----	----	----	----	----
2347	----	----	----	----	----	----	----	----
2350	<10	<10	<5	<5	<5	<2	<10	----
2357	----	----	----	----	----	----	----	----
2363	----	----	----	----	----	----	----	----
2365	----	----	----	----	----	----	----	----
2366	----	----	----	----	----	----	----	----
2369	----	----	----	----	----	----	----	----
2370	<4.00	<2.00	<4.00	<10.0	<10.0	<2.00	<10.0	<10.0
2375	<10	<10	<10	<10	<10	<10	<10	<10
2379	----	----	----	----	----	not detected	----	----
2380	----	----	----	----	----	----	----	----
2385	----	----	----	----	----	----	----	----
2406	not detected	not detected	not detected	----	----	not detected	not detected	----
2464	----	----	9.2	----	6.78	----	----	----
2476	----	----	----	----	----	----	----	----
2492	----	----	----	----	----	----	----	----
2509	not detected	not detected	not detected	----	----	not detected	not detected	----
2553	----	----	----	----	----	----	----	----
2564	----	----	----	----	----	----	----	----
2590	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	----
2624	----	----	----	----	----	----	----	----
2629	<5	<5	<5	<5	<5	<5	<5	<5
2656	not detected	not detected	not detected	detected	not detected	not detected	not detected	not detected
2672	not quantified	not quantified	not quantified	not quantified	not quantified	not quantified	not quantified	not quantified
2678	----	----	----	----	----	----	----	----
2703	----	----	----	----	----	----	----	----
2743	----	----	----	----	----	----	----	----
2789	----	----	----	----	----	----	----	----
2810	----	----	----	----	----	----	----	----
2829	----	----	----	----	----	----	----	----
2851	not detected	4.4811	not detected	not detected	----	not detected	not detected	----
2864	----	----	4.73	----	----	----	----	----
3146	----	----	not detected	----	----	not detected	----	----
3160	----	----	4.99	----	----	----	----	----
3183	----	----	----	----	----	----	----	----

**Other reported Metals in sample #21630-2; results in mg/kg**

lab	Sb	As	Cr	Co	Mn	Ni	Hg	Se	Sr
110	----	----	----	----	----	----	----	----	----
210	----	----	----	----	----	----	----	----	----
348	----	----	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----	----	----
623	not detected	not detected	4351.09	7.91	3898.44	196.62	not detected	not detected	----
840	<10	<10	----	<10	----	----	<5	----	<10
841	<10	<10	<10	<10	<10	15	<10	<10	<10
1126	----	----	----	----	----	----	----	----	----
1910	----	----	----	----	----	----	----	----	----
2115	----	----	2.48	----	----	23.54	----	----	----
2121	----	----	----	----	----	----	----	----	----
2129	----	----	----	----	----	----	----	----	----
2135	----	----	----	----	----	16.235	----	----	----
2159	<10	<5	<10	<10	<10	<10	<5	<10	<10
2184	----	----	2018	----	----	----	<10	----	----
2216	----	----	----	----	----	----	----	----	----
2256	----	----	----	----	----	----	----	----	----
2266	----	----	----	----	----	----	----	----	----
2310	----	----	----	----	----	----	----	----	----
2311	----	----	----	----	----	----	----	----	----
2314	----	----	----	----	----	----	----	----	----
2330	----	----	----	----	----	----	----	----	----
2347	----	----	----	----	----	----	----	----	----
2350	<10	<10	<5	<5	<5	11.58	<2	<10	----
2357	----	----	----	----	----	----	----	----	----
2363	----	----	----	----	----	----	----	----	----
2365	----	----	----	----	----	----	----	----	----
2366	----	----	----	----	----	----	----	----	----
2369	----	----	----	----	----	----	----	----	----
2370	----	----	----	----	----	----	----	----	----
2375	<10	<10	<10	<10	<10	<10	<10	<10	<10
2379	----	----	----	----	----	----	not detected	----	----
2380	----	----	----	----	----	----	----	----	----
2385	----	----	----	----	----	----	----	----	----
2406	not detected	not detected	not detected	----	----	----	not detected	not detected	----
2464	----	----	2576.09	25.26	2391.52	219.03	----	----	----
2476	----	----	----	----	----	----	----	----	----
2492	----	----	----	----	----	----	----	----	----
2509	not detected	not detected	not detected	----	----	----	not detected	not detected	----
2553	----	----	----	----	----	----	----	----	----
2564	----	----	----	----	----	----	----	----	----
2590	<LOQ	<LOQ	<LOQ	<LOQ	----	13.90	<LOQ	<LOQ	----
2624	----	----	----	----	----	----	----	----	----
2629	<5	<5	<5	<5	72.64	17.7	<5	<5	<5
2656	not detected	detected	not detected	detected	not detected	not detected	not detected	not detected	not detected
2672	not quantified	not quantified	not quantified	not quantified	not quantified	13.15	not quantified	not quantified	not quantified
2678	----	----	----	----	----	----	----	----	----
2703	----	----	----	----	----	----	----	----	----
2743	----	----	----	----	----	27.07926	----	----	----
2789	----	----	----	----	----	----	----	----	----
2810	----	----	----	----	----	----	----	----	----
2829	----	----	----	----	----	----	----	----	----
2851	10.4961	14.7972	19.7947	7.0381	----	----	not detected	not detected	----
2864	----	----	----	----	----	----	----	----	----
3146	----	----	2360	----	----	144.5	not detected	----	----
3160	----	----	----	----	----	----	----	----	----
3183	----	----	----	----	----	----	----	----	----

**Other reported Metals in sample #21630-3; results in mg/kg**

lab	Sb	Cd	Pb	Hg	Se	Sr
110	----	not detected	not detected	----	----	----
210	----	----	----	----	----	----
348	----	----	----	----	----	----
551	----	----	----	----	----	----
623	not detected	not detected	25.71	not detected	not detected	----
840	----	----	----	----	----	----
841	----	----	----	----	----	----
1126	----	----	----	----	----	----
1910	----	not detected	not detected	----	----	----
2115	7.37	----	----	----	----	----
2121	----	----	13.8	----	----	----
2129	7.67	<5	<5	<5	<50	<50
2135	----	----	----	----	----	----
2159	<10	<10	15	<10	<10	<10
2184	----	<10	<10	<10	----	----
2216	----	----	----	----	----	----
2256	----	<5	<5	----	----	----
2266	----	----	----	----	----	----
2310	not detected	not detected	not detected	not detected	not detected	not detected
2311	<10	not detected	<10	not detected	not detected	not detected
2314	----	not detected	not detected	----	----	----
2330	----	----	----	----	----	----
2347	----	<5	<20	----	----	----
2350	<10	<0.5	<5	<2	<10	----
2357	----	<5	<20	----	----	----
2363	----	<5.0	<20	----	----	----
2365	----	<5	<5	----	----	----
2366	----	<5	<5	----	----	----
2369	----	<5	<5	----	----	----
2370	----	----	----	----	----	----
2375	<10	<10	<10	<10	<10	<10
2379	----	not detected	not detected	not detected	----	----
2380	----	----	----	----	----	----
2385	----	----	----	----	----	----
2406	not detected	not detected	not detected	not detected	not detected	----
2464	----	----	----	----	----	----
2476	----	----	----	----	----	----
2492	----	----	----	----	----	----
2509	not detected	not detected	26.4064809	not detected	not detected	----
2553	----	----	----	----	----	----
2564	----	not detected	not detected	----	----	----
2590	<LOQ	<LOQ	7.00	<LOQ	<LOQ	----
2624	----	----	----	----	----	----
2629	----	----	----	----	----	----
2656	not detected	not detected	detected	not detected	not detected	not detected
2672	8.220	not quantified	not quantified	not quantified	not quantified	not quantified
2678	----	----	----	----	----	----
2703	----	----	----	----	----	----
2743	----	----	----	----	----	----
2789	----	2.76	20.10	----	----	----
2810	----	----	not detected	----	----	----
2829	----	----	----	----	----	----
2851	23.2209	not detected	13.4816	not detected	not detected	----
2864	----	----	----	----	----	----
3146	----	not detected	not detected	not detected	----	----
3160	----	not detected	7.73	----	----	----
3183	----	5	48	----	----	----

**Other reported Metals in sample #21630-4; results in mg/kg**

lab	Sb	As	Cd	Co	Hg	Se	Sr
110	----	----	not detected	----	----	----	----
210	----	----	----	----	----	----	----
348	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----
623	not detected	not detected	not detected	not detected	not detected	not detected	----
840	----	----	----	----	----	----	----
841	----	----	----	----	----	----	----
1126	----	----	----	----	----	----	----
1910	----	----	not detected	----	----	----	----
2115	----	8.14	----	6.67	----	----	----
2121	----	----	----	----	----	----	----
2129	<5	45.65	<5	38.7	<5	<50	<50
2135	----	----	----	32.26	----	----	----
2159	----	----	----	----	----	----	----
2184	----	----	----	----	----	----	----
2216	----	----	----	----	----	----	----
2256	----	----	<5	----	----	----	----
2266	----	----	----	----	----	----	----
2310	not detected	23.8	not detected	18.6	not detected	not detected	not detected
2311	<10	16.98	not detected	15.68	not detected	not detected	not detected
2314	----	----	not detected	----	----	----	----
2330	----	----	----	----	----	----	----
2347	----	----	----	----	----	----	----
2350	----	----	----	----	----	----	----
2357	----	----	----	----	----	----	----
2363	----	----	----	----	----	----	----
2365	----	----	----	----	----	----	----
2366	----	----	----	----	----	----	----
2369	----	----	----	----	----	----	----
2370	----	----	----	----	----	----	----
2375	----	----	----	----	----	----	----
2379	----	----	not detected	----	not detected	----	----
2380	----	----	----	----	----	----	----
2385	----	----	----	----	----	----	----
2406	not detected	not detected	not detected	----	not detected	not detected	----
2464	----	----	----	----	----	----	----
2476	----	----	----	----	----	----	----
2492	----	----	----	----	----	----	----
2509	not detected	not detected	not detected	----	not detected	not detected	----
2553	----	----	----	----	----	----	----
2564	----	----	not detected	----	----	----	----
2590	----	----	----	----	----	----	----
2624	----	----	----	----	----	----	----
2629	----	----	----	----	----	----	----
2656	not detected	detected	not detected	not detected	not detected	not detected	not detected
2672	not quantified	19.30	not quantified	not quantified	not quantified	not quantified	not quantified
2678	----	----	----	----	----	----	----
2703	----	----	----	----	----	----	----
2743	----	----	----	----	----	----	----
2789	----	----	----	----	----	----	----
2810	----	----	----	----	----	----	----
2829	----	----	----	----	----	----	----
2851	30.1737	61.8789	not detected	not detected	not detected	not detected	----
2864	----	----	----	----	----	----	----
3146	----	----	not detected	----	not detected	----	----
3160	----	----	----	----	----	----	----
3183	----	----	----	----	----	----	----

## APPENDIX 7 Analytical details

lab	Accredited ISO17025	Intake in grams	Digestion acid used	Concentration Acid	Quantify technique used	Remarks
110	---				---	
210	---				---	
348	Yes	0.15	nitric acid	60%	ICP-MS	
551	---				---	
623	Yes	0.2	nitric acid	65	ICP-OES	
840	Yes	0.15	HCL, HNO3	6%HCl:4%HNO3	ICP-OES	
841	---				---	
1126	No	1: 0,1021 2: 0,1023 3: 0,1075	Aqua regia	10 ml Aqua regia up to 100 ml with MilliQ-water	ICP-OES	
1910	No	0,15 g	1 and 4 HNO3 2 and 3 aqua regia	1 and 4 6% HNO3 2 and 3 6% aqua regia	AAS	
2115	---				---	
2121	No	1: 0.0941 2: 0.0942 3: 0.0994 4: 0.0994	HNO3 et HCl	HNO3 : 69.5% HCl : 37%	ICP-MS	
2129	Yes	0,1g	Nitric Acid / Aqua regia	65% HNO3 / 37% HCl	ICP-MS	
2135	Yes	0.2	Nitric acid 65% Hydrochloric acid 37%	Nitric acid 65% HCl 37%	ICP-OES	
2159	Yes	0,2 g	Aqua regia – a mixture of HNO3 and HCl in a ratio of 3:1 (HNO3:HCl)	40 % (v/v) Aqua regia	ICP-MS	
2184	Yes	whole piece lobster ,others 0.1g	HNO3 and HCl	24%	ICP-OES	
2216	Yes	2.8462 g	Nitric acid, Hydrochloric acid	Nitric = 67%, HCl = 37%	ICP-MS	
2256	Yes	0.2651	HNO3 and HCL	69-70% HNO3 36.5-38% HCL	AAS	
2266	No	2.8503	Nitric acid + Hydrochloric acid	57.5%	ICP-MS	
2310	Yes	0.1g	HNO3	65% HNO3	ICP-MS	Reporting limit: <10 mg/kg
2311	Yes	0.1	Nitric Acid	69%	ICP-MS	
2314	No	0.5 gms	Nitric acid	71%	AAS	Reporting Limit is 5 mg/kg
2330	---				---	
2347	Yes				ICP-OES	
2350	Yes	1: 0.2 2: 0.2 3: 0.3/0.2	HCl+HNO3	5%	ICP-OES	
2357	---				---	
2363	Yes	Main:0.4 Tail:0.1 Clasp:0.5	HNO3+HCl		Other,	Analyze technique of Main chain:ICP-OES Analyze technique of Tail chain:AAS Analyze technique of Lobster clasp:AAS
2365	Yes	0.4g	Nitric acid and hydrochloric acid	HNO3-65 % HCl-37 %	ICP-OES	
2366	Yes				ICP-OES	
2369	Yes				AAS	
2370	Yes	0.2 g	HNO3 : 4 mL HCl : 12 mL	HNO3-65 % HCl-37 %	ICP-OES	
2375	Yes	0.15	HNO3-HCl	-	ICP-MS	ISO 16711-1
2379	Yes	0.2 g	Nitric acid + Hydrochloric acid	20 %	Other,	2 analyze technique we used to quantify the metals is ICP-OES and AAS.
2380	Yes	0.1 g	HNO3 & HCl	HNO3-65 % HCl-37 %	ICP-OES	
2385	Yes	2,8613 g (entire sample )	aqua regia (hydrochloric acid/ nitric acid 3/1)	40 mL aqua regia per 100 mL	ICP-OES	Mercury was determined by CV-AAS. Method: ASTM F963-17 Clause 8.3.1 #21630-1: Chain #21630-2: Ring #21630-3: Movable part of Buckle #21630-4: Non-movable part of Buckle
2406	Yes	0.1 g	Nitric acid	3%	ICP-OES	
2464	Yes	approx. 0.2g	Aqua Regia (HCl and HNO3)	Approx. 0.2g / 50 mL	ICP-OES	Method used CPSC-CH-E1001-08.3

lab	Accredited ISO17025	Intake in grams	Digestion acid used	Concentration Acid	Quantify technique used	Remarks
2476	---				---	
2492	Yes	0.125 g	HNO3 and HCl	65% & 37%	ICP-MS	
2509	Yes	0.1250 g	Nitric Acid + Hydrochloric Acid	Nitric Acid (26%) + HCl (65 – 69%)	ICP-OES	
2553	Yes	0.2011g	Aqua Regia	Hydrochloric Acid : Nitric Acid = 3 : 1	Other,	ICP-MS followed by ICP-OES
2564	Yes		Nitric Acid	65% Nitric Acid	AAS	
2590	Yes	0.1	nitric acid and hydrochloric acid	8 %	ICP-MS	method used EN 16711-1
2624	Yes	0.2 g	HNO3	20%	AAS	
2629	Yes	0.3g	Aqua regia		ICP-OES	
2656	No	-	-	-	XRF	surfacic analysis - not quantitative
2672	Yes	0,1	HNO3	approx 33	ICP-OES	
2678	---				---	
2703	No	0.1930g	Nitric acid & Hydrochloric acid	Concentrated aqua regia (65% Nitric acid/ 37% HCl)	ICP-OES	A combined sample of all parts of the necklace was used
2743	Yes	0,05	HNO3+HCl	HNO3 (65%), HCl (37%)	ICP-OES	
2789	Yes	0.15	nitric acid + hydrochloric acid	11%	ICP-MS	
2810	Yes	1: 0,53g 2: 1,06g 3: 0,06g 4: 0,38g	Nitric acid (65%) + Hydrochloric acid (37%)	approximately 50%	ICP-OES	
2829	Yes	about 100 mg	Nitric Acid and Hydrochloridric acid		ICP-AES	
2851	Yes	0.2 grams	Nitric Acid and Hydrochloridric acid	65% / 37%	ICP-OES	
2864	Yes	0.1 g	Aqua Regia	70% HNO3 37% HCL	ICP-OES	
3146	Yes	Ball: 0.8237 carabiner : 0.5370 Extensio n: 0.4186 Connecti ng: 0.2233	Nitric acid p. a. Hydrochloric acid p. a.	Nitric acid (65 %) Hydrochloric acid (25 %)	ICP-MS	
3160	Yes	0,1g	HNO3 and HCl	6,5%	ICP-MS	Method based on CPSC-CH-E1001-8.3
3183	---				---	



## APPENDIX 8

### Number of participants per country

1 lab in BANGLADESH  
1 lab in BRAZIL  
1 lab in CAMBODIA  
1 lab in CANADA  
1 lab in EGYPT  
3 labs in FRANCE  
6 labs in GERMANY  
4 labs in HONG KONG  
3 labs in INDIA  
1 lab in INDONESIA  
6 labs in ITALY  
1 lab in MALAYSIA  
1 lab in MOROCCO  
7 labs in P.R. of CHINA  
1 lab in POLAND  
1 lab in SOUTH KOREA  
3 labs in SPAIN  
1 lab in SRI LANKA  
3 labs in TAIWAN  
1 lab in THAILAND  
1 lab in THE NETHERLANDS  
1 lab in TUNISIA  
2 labs in TURKEY  
2 labs in U.S.A.  
1 lab in UNITED KINGDOM  
3 labs in VIETNAM

## APPENDIX 9

### 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

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ISO5725:86
- 3 ISO5725 parts 1-6:94
- 4 ISO13528:05
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- 6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 8 J.N. Miller, Analyst, 118, 455, (1993)
- 9 Analytical Methods Committee, Technical Brief, No 4, January 2001
- 10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
- 11 W. Horwitz and R. Albert, J. AOAC Int, 79, 3, 589-621, (1996)
- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
- 13 Regulation (EC) No. 1907/2006, December 2006, REACH, amending Directive 1999/45/EC
- 14 Consumer Product Safety Improvement Act of 2008, Public Law 110-214, Aug14, 2008