



Institute for
Interlaboratory Studies

**Results of Proficiency Test
Migration of elements EN71-3
Category II
April 2023**

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Correctors: ing. G.A. Oosterlaken-Buijs & ing. M. Meijer
Approved by: ing. A.S. Noordman-de Neef

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1 INTRODUCTION

Toy safety is the practice of ensuring that toys, especially those made for children, are safe usually through the application of set safety standards. In many countries, toys must be able to pass safety tests in order to be sold. Many regions model their safety standards on the EU's EN71 standard, either directly, or through adoption of the ISO8124-3 standard which in itself is modelled on EN71. In Europe, toys must meet the criteria set by the EC Toy Safety Directive 2009/48/EC which applies to toy imports into the EU since 20th of July 2011. There is an exception for the chemical requirements under part III of Annex II of this directive. These chemical requirements came into force on 20th of July 2013. The test methods EN71-3:19+A1:21 and ISO8124-3:20 both describe the determination of Migration of elements (metals that are considered hazardous) when a toy gets into contact with an acid solution (0.07 n HCl, simulating a gastric acid solution).

Since 2010 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Migration of elements EN71-3 every year. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of the Migration of elements. This proficiency test describes the Migration of elements EN71-3 for category II samples.

In this interlaboratory study 41 laboratories in 16 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Migration of elements EN71-3 for category II 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 sample of 10 mL fingerprint labelled #23560. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

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

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of white fingerprint was purchased in a local shop. This batch was artificially fortified with some elements. After homogenization 50 PE bottles of 10 mL were filled and labelled #23560.

The homogeneity of the subsamples was checked by determination of Cadmium as Cd and Copper as Cu in accordance with EN71-3 on 8 stratified randomly selected subsamples.

	Cadmium as Cd in mg/kg	Copper as Cu in mg/kg
sample #23560-1	2.480	77.30
sample #23560-2	2.350	68.20
sample #23560-3	2.720	68.30
sample #23560-4	2.550	69.60
sample #23560-5	2.300	69.00
sample #23560-6	2.650	71.90
sample #23560-7	2.340	68.80
sample #23560-8	2.343	68.38

Table 1: homogeneity test results of subsamples #23560

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

	Cadmium as Cd in mg/kg	Copper as Cu in mg/kg
r (observed)	0.446	8.73
reference test method	EN71-3:19+A1:21	EN71-3:19+A1:21
0.3 x R (reference test method)	0.414	8.84

Table 2: evaluation of the repeatabilities of subsamples #23560

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample of fingerprint labelled #23560 was sent on March 22, 2023.

2.5 ANALYZES

The participants were requested to determine the migration of nineteen elements (Aluminum, Antimony, Arsenic, Barium, Boron, Cadmium, Chromium (III), Chromium (VI), Cobalt, Copper, Lead, Manganese, Mercury, Nickel, Selenium, Strontium, Tin, Organic Tin and Zinc). It was also 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.

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

3 RESULTS

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

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

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

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

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

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

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

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

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

3.3 Z-SCORES

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

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

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

The z-scores were calculated according to:

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

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. 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. Two participants reported test results after the final reporting date and two other participants did not report any test results. Not all participants were able to report all elements requested. In total 39 participants reported 149 numerical test results. Observed were 9 outlying test results, which is 6.0%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER ELEMENT

In this section the reported test results are discussed per element. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

Test method EN71-3:19+A1:21 is considered to be the official test method for the determination of migration of elements analyzed in different matrices. The precision data in EN71-3:19+A1:21 are given in Table 4 and in appendix Table C.1. Table 4 contains precision data from an interlaboratory study. The committee was not able to obtain precision data for all elements for each category via an interlaboratory study. In order to compensate for missing data for certain element and category combinations estimations for the reproducibility have been considered by the committee based on table 4 and input from experts. These precision data are given in table C.1 and are used to evaluate the performance of the group of participants in this PT.

In EN71-3:19+A1:21 is mentioned that maintaining the pH between 1.1 and 1.3 is very important for the determination of the migration of elements. Based on the answers reported one participant reported pH value outside the range of 1.1 and 1.3. The reported test value appeared a statistical outlier and therefore no test results need to be excluded from the statistical evaluations.

Aluminum as Al: This determination was problematic for a number of participants. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN71-3:19+A1:21.

Cadmium as Cd: This determination was not problematic. Two statistical outliers were observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN71-3:19+A1:21.

Copper as Cu: 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 EN71-3:19+A1:21.

Lead as Pb: 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 EN71-3:19+A1:21.

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 results can be found in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Element	unit	n	average	2.8 * sd	R(lit)
Aluminum as Al	mg/kg	34	110.8	23.7	46.6
Cadmium as Cd	mg/kg	35	2.30	0.40	1.29
Copper as Cu	mg/kg	35	62.1	11.0	26.1
Lead as Pb	mg/kg	35	3.04	0.80	1.70

Table 3: reproducibilities of tests on sample #23560

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

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

	April 2023	April 2022	April 2021	April 2020	April 2019
Number of reporting laboratories	39	27	27	36	37
Number of test results	149	72	94	101	99
Number of statistical outliers	9	1	5	8	11
Percentage of statistical outliers	6.0%	1.4%	5.3%	7.9%	11.1%

Table 4: 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	April 2023	April 2022	April 2021	April 2020	April 2019	Target
Aluminum	8%	---	---	9%	---	15%
Antimony	---	---	---	---	---	30%
Arsenic	---	---	---	---	---	20%
Barium	---	9%	---	---	22%	15%
Boron	---	---	9%	---	---	15%
Cadmium	6%	7%	---	6%	---	20%

Element	April 2023	April 2022	April 2021	April 2020	April 2019	Target
Chromium (III)	---	---	25%	---	---	20%
Chromium (VI)	---	---	---	---	---	50%
Cobalt	---	---	8%	---	---	20%
Copper	6%	---	---	---	---	15%
Lead	9%	---	---	6%	22%	20%
Manganese	---	---	---	---	---	15%
Mercury	---	---	---	---	---	30%
Nickel	---	7%	6%	---	19%	20%
Selenium	---	---	---	---	---	20%
Strontium	---	---	---	---	---	15%
Tin	---	---	---	---	---	20%
Organic Tin	---	---	---	---	---	50%
Zinc	---	---	---	---	---	15%

Table 5: development of the uncertainties over the years

The uncertainties observed in this PT are comparable with the uncertainties observed in the 2020 PT and in the 2022 PT.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

Almost all reporting participants mentioned that they are ISO/IEC17025 accredited for the category II determination of Migration of elements EN71-3.

Furthermore, the participants were asked to provide some analytical details which are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

- 76% of the participants used a sample intake between 0.1 to 0.4 grams and 24% used a sample intake between 0.5 to 1 gram.
- Almost all participants have used a solution with a pH between 1.1 and 1.3 (with or without adjustment of HCL solution) for the determination of the elements.
- Almost all participants mentioned to have used a volume ratio of 5 mL of HCl solution per 100 mg sample intake for the migration.

As the majority of the group follow the same analytical procedures and the target reproducibilities are met no further analysis to the analytical details has been done.

5 DISCUSSION

It appeared that EN71-3:19+A1:21 has been followed well by most of the participants. One participant has used a solution with a pH above 1.3 to measure the metals and three participants did not use the correct volume ratio. The effect of incorrect volume ratio used on the determination is neglectable as all reported test values have good z-scores.

When the results of this interlaboratory study are compared to the Migration limits from toy materials for category II as mentioned in EN71-3:19+A1:21 (see table below) it was noticed that all participants would have made identical decisions about the acceptability of the material for the determined elements. Almost all reporting participants would have rejected sample #23560 for too high level of Cadmium and/or Lead. All participants would have accepted the sample when only Aluminum or Copper are considered.

Element	Category II mg/kg
Aluminum	560
Antimony	11.3
Arsenic	0.9
Barium	375
Boron	300
Cadmium	0.3
Chromium (III)	9.4
Chromium (VI)	0.005
Cobalt	2.6
Copper	156
Lead	0.5
Manganese	300
Mercury	1.9
Nickel	18.8
Selenium	9.4
Strontium	1125
Tin	3750
Organic Tin	0.2
Zinc	938

Table 6: Migration limits from toy materials for Category II as mentioned in EN71-3:19+A1:21

6 CONCLUSION

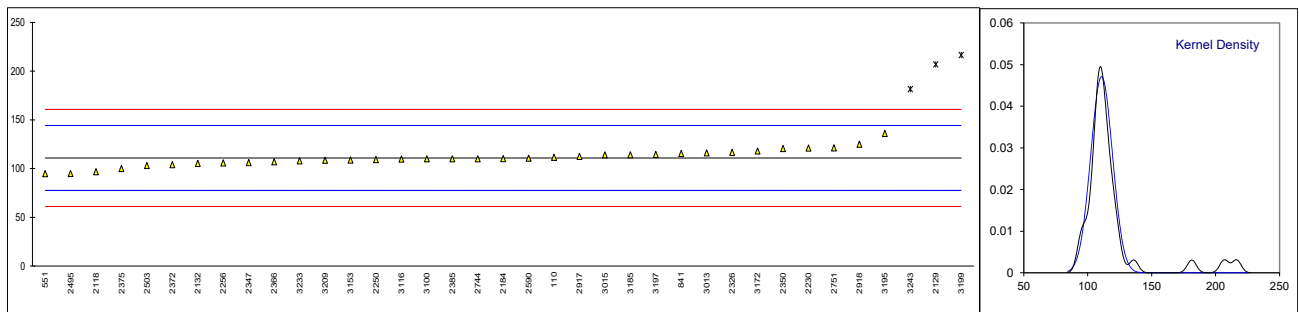
In this PT it appeared that version of EN71-3:19+A1:21 has been followed well by most of the participants. Most of the participants had detected the elements correctly in the samples. Although it can be concluded that most of the participants have no problems with the determination migration of elements in the sample of this PT each participant will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of migration of Aluminum as Al on fingerprint sample #23560; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	EN71-3	111.515		0.04	
551	EN71-3	94.773		-0.97	
841	EN71-3	115.46		0.28	
2118	EN71-3	96.62		-0.86	
2129	EN71-3	206.833	R(0.01)	5.77	
2132	EN71-3	105.32		-0.33	
2184	EN71-3	110.2		-0.04	
2230	EN71-3	121		0.61	
2250	EN71-3	109.3		-0.09	
2256	EN71-3	105.78		-0.30	
2326	EN71-3	116.58		0.35	
2347	EN71-3	106		-0.29	
2350	EN71-3	120.58		0.59	
2366	EN71-3	107		-0.23	
2372	EN71-3	104		-0.41	
2375	EN71-3	100		-0.65	
2385	EN71-3	110		-0.05	
2495	EN71-3	94.826		-0.96	
2503	EN71-3	103		-0.47	
2590	EN71-3	110.65		-0.01	
2744	EN71-3	110		-0.05	
2751	EN71-3	121.10		0.62	
2917	EN71-3	112.38		0.09	
2918	EN71-3	125		0.85	
2984		----		----	
3013	EN71-3	116		0.31	
3015	EN71-3	114		0.19	
3100	EN71-3	109.93		-0.05	
3116		109.70		-0.07	
3124		----		----	
3153	EN71-3	108.84		-0.12	
3172	EN71-3	118		0.43	
3185	EN71-3	114.12		0.20	
3195	EN71-3	136		1.51	
3197	EN71-3	114.48		0.22	
3199	EN71-3	216.49	R(0.01)	6.35	
3209	EN71-3	108.49		-0.14	
3233	EN71-3	107.93		-0.18	
3243	EN71-3	181.41	C,R(0.01)	4.24	First reported 12097.057
3248		----		----	
8005		----		----	

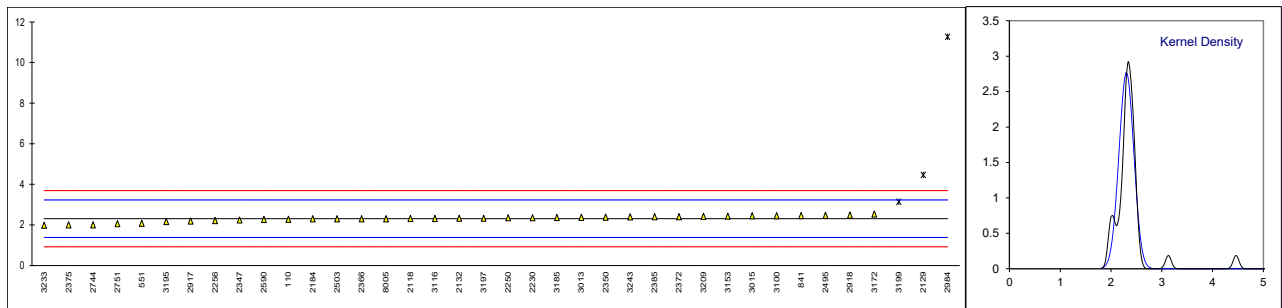
normality suspect
 n 34
 outliers 3
 mean (n) 110.8404
 st.dev. (n) 8.46929 RSD = 8%
 R(calc.) 23.7140
 st.dev.(EN71-3:19+ A1:21) 16.62606
 R(EN71-3:19+A1:21) 46.5530



Determination of migration of Cadmium as Cd on fingerprint sample #23560; results in mg/kg

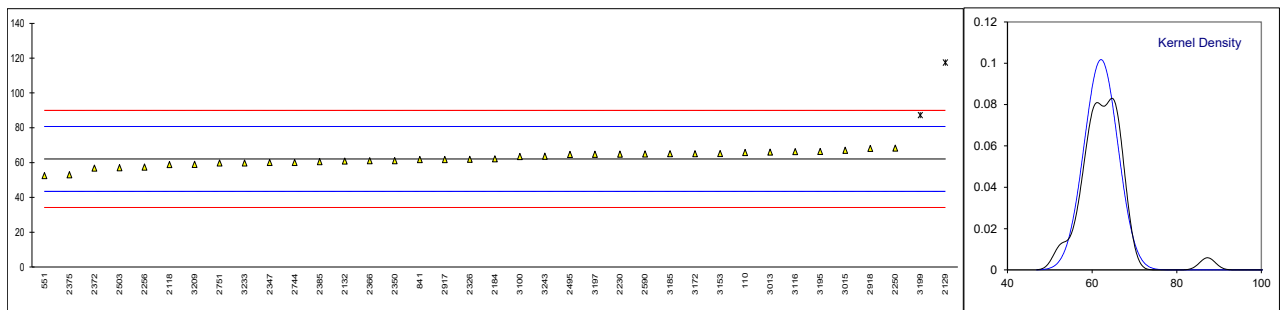
lab	method	value	mark	z(targ)	remarks
110	EN71-3	2.280		-0.05	
551	EN71-3	2.079		-0.49	
841	EN71-3	2.46		0.34	
2118	EN71-3	2.31		0.01	
2129	EN71-3	4.463	R(0.01)	4.68	
2132	EN71-3	2.33		0.06	
2184	EN71-3	2.30		-0.01	
2230	EN71-3	2.35		0.10	
2250	EN71-3	2.348		0.09	
2256	EN71-3	2.21		-0.20	
2326	EN71-3	ND		----	
2347	EN71-3	2.24		-0.14	
2350	EN71-3	2.38		0.16	
2366	EN71-3	2.3		-0.01	
2372	EN71-3	2.4		0.21	
2375	EN71-3	2		-0.66	
2385	EN71-3	2.4		0.21	
2495	EN71-3	2.474		0.37	
2503	EN71-3	2.3		-0.01	
2590	EN71-3	2.27		-0.07	
2744	EN71-3	2.0		-0.66	
2751	EN71-3	2.06		-0.53	
2917	EN71-3	2.19		-0.25	
2918	EN71-3	2.48		0.38	
2984	ISO8124-3	11.2644	C,R(0.01)	19.44	First reported 9.025
3013	EN71-3	2.37		0.14	
3015	EN71-3	2.45		0.32	
3100	EN71-3	2.45		0.32	
3116		2.31		0.01	
3124		----		----	
3153	EN71-3	2.43		0.27	
3172	EN71-3	2.54		0.51	
3185	EN71-3	2.36		0.12	
3195	EN71-3	2.16		-0.31	
3197	EN71-3	2.33		0.06	
3199	EN71-3	3.13	ex	1.79	Test result excluded, see below.
3209	EN71-3	2.42		0.25	
3233	EN71-3	1.98		-0.70	
3243	EN71-3	2.393	C	0.19	First reported 254.323
3248		----		----	
8005		2.30		-0.01	
normality		OK			
n		35			
outliers		2 +1ex			
mean (n)		2.3044			
st.dev. (n)		0.14404	RSD = 6%		
R(calc.)		0.4033			
st.dev.(EN71-3:19+A1:21)		0.46088			
R(EN71-3:19+A1:21)		1.2905			

Lab 3199: the test result was excluded for statistical evaluation as 3 of the 4 evaluated test results are statistical outliers



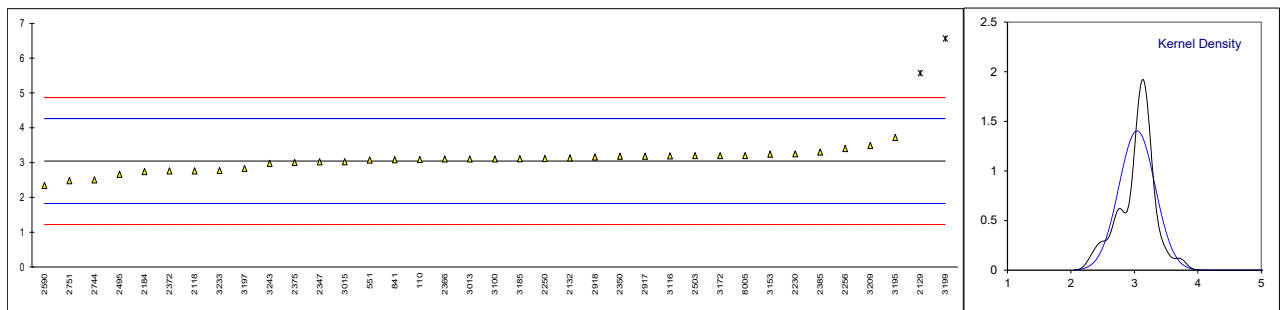
Determination of migration of Copper as Cu on fingerprint sample #23560; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	EN71-3	65.780		0.39	
551	EN71-3	52.415		-1.04	
841	EN71-3	61.69		-0.04	
2118	EN71-3	58.79		-0.36	
2129	EN71-3	117.424	R(0.01)	5.94	
2132	EN71-3	60.86		-0.13	
2184	EN71-3	62.12		0.00	
2230	EN71-3	64.8		0.29	
2250	EN71-3	68.29		0.66	
2256	EN71-3	57.37		-0.51	
2326	EN71-3	61.87		-0.03	
2347	EN71-3	60		-0.23	
2350	EN71-3	61.07		-0.11	
2366	EN71-3	61		-0.12	
2372	EN71-3	56.8		-0.57	
2375	EN71-3	53		-0.98	
2385	EN71-3	60.5		-0.17	
2495	EN71-3	64.606		0.27	
2503	EN71-3	57		-0.55	
2590	EN71-3	64.92		0.30	
2744	EN71-3	60		-0.23	
2751	EN71-3	59.70		-0.26	
2917	EN71-3	61.71		-0.04	
2918	EN71-3	68.1		0.64	
2984		----		----	
3013	EN71-3	66		0.42	
3015	EN71-3	67		0.53	
3100	EN71-3	63.43		0.14	
3116		66.25		0.44	
3124		----		----	
3153	EN71-3	65.18		0.33	
3172	EN71-3	65.1		0.32	
3185	EN71-3	65.08		0.32	
3195	EN71-3	66.3		0.45	
3197	EN71-3	64.72		0.28	
3199	EN71-3	87.27	R(0.01)	2.70	
3209	EN71-3	58.94		-0.34	
3233	EN71-3	59.71		-0.26	
3243	EN71-3	63.629	C	0.16	First reported 6758.416
3248		----		----	
8005		----		----	
normality		OK			
n		35			
outliers		2			
mean (n)		62.1066			
st.dev. (n)		3.92150	RSD = 6%		
R(calc.)		10.9802			
st.dev.(EN71-3;19+A1:21)		9.31599			
R(EN71-3;19+A1:21)		26.0848			



Determination of migration of Lead as Pb on fingerprint sample #23560; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	EN71-3	3.090		0.08	
551	EN71-3	3.073		0.05	
841	EN71-3	3.08		0.06	
2118	EN71-3	2.76		-0.46	
2129	EN71-3	5.569	R(0.01)	4.15	
2132	EN71-3	3.13		0.14	
2184	EN71-3	2.74		-0.50	
2230	EN71-3	3.25		0.34	
2250	EN71-3	3.118		0.13	
2256	EN71-3	3.40		0.59	
2326	EN71-3	ND		----	
2347	EN71-3	3.02		-0.04	
2350	EN71-3	3.18		0.23	
2366	EN71-3	3.1		0.10	
2372	EN71-3	2.76		-0.46	
2375	EN71-3	3		-0.07	
2385	EN71-3	3.3		0.42	
2495	EN71-3	2.659		-0.63	
2503	EN71-3	3.2		0.26	
2590	EN71-3	2.34		-1.15	
2744	EN71-3	2.5		-0.89	
2751	EN71-3	2.48		-0.92	
2917	EN71-3	3.18		0.23	
2918	EN71-3	3.16		0.19	
2984	ISO8124-3	not detected		----	possibly a false negative test result?
3013	EN71-3	3.1		0.10	
3015	EN71-3	3.02		-0.04	
3100	EN71-3	3.10		0.10	
3116		3.19		0.24	
3124		----		----	
3153	EN71-3	3.24		0.33	
3172	EN71-3	3.2		0.26	
3185	EN71-3	3.11		0.11	
3195	EN71-3	3.72		1.11	
3197	EN71-3	2.83		-0.35	
3199	EN71-3	6.56	R(0.01)	5.78	
3209	EN71-3	3.49		0.74	
3233	EN71-3	2.77		-0.45	
3243	EN71-3	2.976	C	-0.11	First reported 329.987
3248		----		----	
8005		3.20		0.26	
normality		OK			
n		35			
outliers		2			
mean (n)		3.0419			
st.dev. (n)		0.28412	RSD = 9%		
R(calc.)		0.7955			
st.dev.(EN71-3;19+A1:21)		0.60838			
R(EN71-3;19+A1:21)		1.7035			



APPENDIX 2

Determination of migration of other elements on sample #23560; results in mg/kg

lab	Sb	As	Ba	B	Cr (III)	Cr(VI)	Co	Mn
110	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
551	0.02378	0.0668	0.0841	-----	0.2974	-----	-----	0.1125
841	<0.1	<0.1	<2.5	<2.5	0.020	<0.002	<0.1	<2.5
2118	0.12	0.13	0.05	0.33	0.01	0	0	0.08
2129	<1,0	<0,10	<10	<10	0.014	not tested	<0,10	<10
2132	<1	<0.05	<25	<25	<1	<0.0035	<0.1	<25
2184	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2230	-----	-----	-----	-----	-----	-----	-----	-----
2250	not detected	not detected	not detected	not detected	not detected	<0,005	not detected	not detected
2256	<2.00000	<0.15000	<2.00000	3.31	0.026	ND	<1.00000	<2.00000
2326	ND	ND	ND	ND	ND	ND	ND	ND
2347	<1.0	<0.4	<50	<50	<1.0	<0.0025	<0.5	<50
2350	<1	<0.4	<50	<50	<1	<0.0025	<0.5	<50
2366	<1.0	<0.4	<50	<50	<1.0	<0.025	<0.5	<50
2372	<1	<0.4	<50	<50	<1	0.0069	<0.5	<50
2375	<1	<0.4	<50	<50	<1	<0.0025	<0.5	<50
2385	<5	<1	<1	<1	<1	<0.005	<1	<5
2495	<1	<0.5	<5	<5	<1	-----	<0.5	<5
2503	-----	2.0	-----	2.2	-----	-----	-----	-----
2590	< L.O.Q.	< L.O.Q.	< L.O.Q.	-----	< L.O.Q.	-----	< L.O.Q.	< L.O.Q.
2744	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2751	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2917	<1.0	0.58	<0.5	2.62	<0.5	-----	<0.5	<0.5
2918	not detected	not detected	not tested	not tested	not detected	-----	not tested	not tested
2984	not detected	not detected	not detected	-----	not analyzed	not analyzed	-----	-----
3013	not detected	not detected	0.11	0.31	1.0	0.1013	not detected	not detected
3015	<1	<0.5	<10	<50	<1	<0.0025	<1	<10
3100	<1.0	<0.5	<10	<50	<1	<0.0025	<1.0	<10
3116	<2	<0.15	<2	<5	<1	<0.002	<2	<2
3124	-----	-----	-----	-----	-----	-----	-----	-----
3153	< 1	< 0.3	< 10	< 50	< 1	< 0.01	< 1	< 10
3172	< 2	< 0.1	< 10	< 50	< 2	< 0.005	< 1	< 50
3185	<1	<0.5	<10	<50	<1	<0.0025	<1	<10
3195	<0,50	<0,20	<5,0	<5,0	not analyzed	not analyzed	<0,50	<5,0
3197	<1	<0.5	<10	<10	<1	<0.0025	<1	<10
3199	Not detected	Not detected	Not detected	Not detected	0.033	Not tested	Not detected	Not detected
3209	<1.0	<0.5	<1.0	<1.0	<1.0	<0.0025	<1.0	<1.0
3233	< 5	< 0.05	< 5	< 5	0.01	-----	< 0.5	< 5
3243	2.081	1.530	2102.899	6.132	1.296	0	0.408	90.533
3248	-----	-----	-----	-----	-----	-----	-----	-----
8005	<2	<2	<2	-----	-----	-----	-----	-----

Determination of migration of other elements on sample #23560; results in mg/kg ---continued---

lab	Hg	Ni	Se	Sr	Sn	Org Sn	Zn
110	not detected	not detected	not detected	not detected	not detected	not analyzed	24.519
551	----	0.2397	0.0826	0.2116	----	----	19.660
841	<0.1	<2.5	<0.5	<2.5	<0.025	<0.04	<2.5
2118	0.01	0.26	0	0.15	0.02	not analyzed	0.23
2129	<0,10	1.078	<1,0	<100	0.1	not tested	<10
2132	<0.1	<1	<1	<50	<10	Not detected	<50
2184	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2230	----	----	----	----	----	----	----
2250	not detected	not detected	not detected	not detected	not detected	<0,1	not detected
2256	<0.02000	0.23	<2.00000	<2.00000	0.02	ND	<2.00000
2326	ND	ND	ND	ND	ND	ND	ND
2347	<0.5	<9.0	<4.0	<50	<0.08	<0.08	<50
2350	<0.5	<9	<4	<50	<0.08	<0.03	<50
2366	<0.5	<9	<4	<50	<0.03	<0.03	<50
2372	<0.5	<9	<4	<50	<4.9	not analyzed	<50
2375	<0.5	<9	<4	<50	<0.08	----	<50
2385	<1	<1	<1	<5	<5	<0.246	<5
2495	<0.5	<1	<1	<5	<5	----	<5
2503	----	----	----	----	----	----	2.3
2590	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	----	1.09
2744	not detected	0.9	not detected	not detected	3.5	not detected	20
2751	Not Detected	0.95	Not Detected	Not Detected	3.35	Not Detected	21.25
2917	<0.01	<0.5	<0.3	<0.5	<0.5	----	<0.5
2918	not detected	0.32	not detected	0.15	not tested	----	0.90
2984	not detected	----	not detected	----	----	----	----
3013	not detected	0.94	not detected	0.24	not detected	----	0.62
3015	<1	<1	<1	<100	<0.05	<0.15	<100
3100	<1.0	<1.0	<1.0	<100	<0.05	<0.1	<100
3116	<0.15	<2	<2	<2	<2	<0.04	<5
3124	----	----	----	----	----	----	----
3153	< 0.5	< 1	< 1	< 100	< 10	< 0.1	< 100
3172	< 0.5	< 5	< 1	< 50	< 50	----	< 50
3185	<1	<1	<1	<100	<0.05	<0.15	<100
3195	<0,1	<2,0	0.586	<5,0	<0,10	not analyzed	<5,0
3197	<0.5	<1	<1	<10	<10	<0.02	<10
3199	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested	Not detected
3209	<1.0	<1.0	<1.0	<1.0	<0.05	<0.2	<1.0
3233	< 0.5	< 5	< 5	< 5	< 0.05	----	< 5
3243	0.129	0.272	0.227	198.294	6.299	----	59.785
3248	----	----	----	----	----	----	----
8005	<2	----	<2	----	----	----	----

APPENDIX 3 Analytical details

lab	ISO/IEC17025 accredited	Sample intake (g)	Amount of 0.07 mol/L HCl solution used (mL)	pH after shaking	pH adjusted after shaking?	pH after adjustment
110	Yes	0.25 g	12.5	1.3	No	
551	Yes	0.25g	12.5mL	1.5	Yes	1.12
841	---	0.2	10ml	1.18	No	
2118	Yes	1 g	50 ml	1.37	Yes	1.19
2129	Yes	0,5g	25ml	1,1	No	1,11
2132	Yes	0.2002g	10ml	1.20	No	
2184	Yes	0.2g	10ml	1.28	Yes	
2230	Yes	0.1022g	5	1.3	No	
2250	Yes	0,2	10	not noted	Yes	not noted
2256	Yes	0.1530	7.65	1.298	No	
2326	Yes	0.5228 GM	26.1 ML	1.1 - 1.2	No	
2347	Yes	0.3g	15ml	1.21	No	
2350	Yes	1.0079g	50 mL	1.2	No	
2366	---				---	
2372	Yes	0.5250 g	25 ml	1.140	Yes	1.185
2375	Yes	0.1 gram	5 mL	1,18	No	
2385	Yes	~0.4	Factor 50	1.23	No	
2495	Yes	0.12	6.0	1.15	No	
2503	Yes	0,1812			---	
2590	Yes	0.1	10	1.61	Yes	1.14
2744	Yes	0,1	5	1,20	No	1,20
2751	Yes	0,0990	50	1,39	Yes	1,25
2917	Yes	1.0	50	1.34	Yes	1.08
2918	Yes	0,2022/0,2090/0,2063	10,115/10,450/10,315	1,066/1,101/1,071	No	
2984	Yes	0.2001	10	1.4	No	-
3013	Yes	0,8240	41		---	
3015	Yes	0.1	5	1.2	No	-
3100	Yes	0.2g	10mL	1.25	No	No
3116	Yes	0.5	25		---	
3124	---				---	
3153	Yes	0.2 gram	10 mL	1.20	No	N/A
3172	Yes				---	
3185	Yes	0.2523g	12.62mL	1.23	No	
3195	No	0,25	12,5	1,2	No	1,2
3197	Yes	0.1511	7.5	1.28	Yes	1.19
3199	Yes	0.1045 g	5.2 mL	1.2	No	
3209	Yes	0.2006	10	1.27	No	1.27
3233	Yes	0.1046g	5.2	1.28	Yes	1.12
3243	Yes	0,6 g	30,5 mL		No	
3248	---				---	
8005	Yes	0.1	5		---	

APPENDIX 4

Number of participants per country

1 lab in BELGIUM
1 lab in BRAZIL
1 lab in DENMARK
1 lab in FRANCE
7 labs in GERMANY
6 labs in HONG KONG
1 lab in INDONESIA
3 labs in ITALY
1 lab in KOREA, Republic of
7 labs in P.R. of CHINA
1 lab in PAKISTAN
1 lab in SLOVENIA
2 labs in TAIWAN
4 labs in TURKEY
3 labs in U.S.A.
1 lab in VIETNAM

APPENDIX 5

Abbreviations

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

Literature

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