Institute for
Interlaboratory Studies

Results of Proficiency Test
Migration of elements EN71-3
Category 1
April 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. C.M. Nijssen-Wester
Correctors: ing. R.J. Starink & ing. M. Meijer
Approved by: ing. A.S. Noordman-de Neef

Report: iis22V02A

June 2022

CONTENTS

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 Migration of elements EN71-3 every year. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test for the determination of Migration of elements. This proficiency test describes the Migration of elements EN71-3 for category 1 samples.

In the interlaboratory study 33 laboratories in 15 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of this Migration of elements EN71-3 for category 1 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 approximately 0.5 grams of plaster labelled #22550.
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 Organization, 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 off-white plaster fortified with heavy metals was selected. After homogenization 60 plastic bags were filled with approximately 0.5 grams each and labelled #22550.
The batch for sample #22550 was used in a previous proficiency test on Migration of elements as sample #13042 in iis13V02. Therefore, homogeneity of the subsamples was assumed.
To each of the participating laboratories one plaster sample of 0.5 grams labelled #22550 was sent on March 2, 2022.

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) applying the analysis procedure that is routinely used in the laboratory. It was also requested to report if the laboratory was accredited for the determination Migration of 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:

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

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

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

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

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. Two participants reported test results after the extended reporting date and two other participants did not report any test results. Not all participants were able to report all tests requested.
In total 31 reporting laboratories submitted 167 numerical test results. Observed were 3 outlying test results, which is 1.8%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

All data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER 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.

EN71-3 method is considered to be the official test method for the determination of elements migrated from different matrices. In 2019 a new version of EN71-3 is published. In 2021 an amended version of the method is published in which the most significant change is the lower limits published in Table 2 for Aluminum.
In test method of EN71-3:19+A1:21 precision data are given in Table 4 and in 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 a part is introduced that maintaining the pH between 1.1 and 1.3 is very important for the determination of the migration of elements. Therefore, based on the answers given by the participants, none of the participants reported pH values outside the range of 1.1 and 1.3 and there is no need to exclude test results from the statistical evaluations.

Aluminum: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN71-3:19+A1:21.

Copper: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not agreement with the requirements of EN71-3:19+A1:21.

Lead: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN71-3:19+A1:21.

Manganese: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN71-3:19+A1:21.

Strontium: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN71-3:19+A1:21.

Zinc: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN71-3:19+A1:21.

The majority of the participants agreed on a concentration near or below the limit of detection for all other reported 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 reproducibilities as declared by the reference test method and the reproducibilities as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from the reference method are presented in the next table.

Element	unit	n	average	2.8 * sd	R(lit)
Aluminum as Al	mg/kg	27	331	119	139
Copper as Cu	mg/kg	27	2507	1610	1053
Lead as Pb	mg/kg	30	137	49	77
Manganese as Mn	mg/kg	27	66.6	21.3	28.0
Strontium as Sr	mg/kg	27	333	200	140
Zinc as Zn	mg/kg	26	6092	2487	2559

Table 1: reproducibilities of tests on sample #22550

Without further statistical calculations it can be concluded that for many tests there is a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF APRIL 2022 WITH PREVIOUS PTS

	April 2022	April 2021	April 2020	April 2019	April 2018
Number of reporting laboratories	31	38	37	36	91
Number of test results	167	197	243	123	449
Number of statistical outliers	3	11	4	8	5
Percentage of statistical outliers	1.8%	5.6%	1.6%	6.5%	11%

Table 2: comparison with previous proficiency tests

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

The uncertainties determined in this PT are compared with the relative standard deviations as found in previous years and with the target requirements in the next table.

Element	April 2022	April 2021	April 2020	April 2019	2013-2018	Target
Aluminum	13%	34%	17%	29%	20-23%	15%
Antimony	n.e.	15%	n.e.	n.e.	28-47%	30%
Arsenic	n.e.	13%	n.e.	n.e.	32%	20%
Barium	n.e.	n.e.	n.e.	n.e.	20%	20%
Boron	n.e.	n.e.	n.e.	n.e.	n.e.	15%
Cadmium	n.e.	14%	14%	n.e.	18%	20%
Chromium (III)	n.e.	n.e.	n.e.	n.e.	44%	15%

Element	April 2022	April 2021	April 2020	April 2019	2013-2018	Target
Chromium (VI)	n.e.	n.e.	n.e.	n.e.	n.e.	50%
Cobalt	n.e.	n.e.	n.e.	n.e.	n.e.	15%
Copper	23%	n.e.	n.e.	10%	17-28%	15%
Lead	13%	17%	23%	26%	18-30%	20%
Manganese	11%	n.e.	10%	n.e.	12-16%	15%
Mercury	n.e.	n.e.	n.e.	n.e.	n.e.	30%
Nickel	n.e.	10%	n.e.	n.e.	15%	20%
Selenium	n.e.	n.e.	n.e.	n.e.	25%	15%
Strontium	21%	10%	12%	20%	15-21%	15%
Tin	n.e.	n.e.	n.e.	n.e.	n.e.	20%
Organic Tin	n.e.	n.e.	n.e.	n.e.	n.e.	50%
Zinc	15%	n.e.	12%	n.e.	14-17%	15%

Table 3: development of uncertainties over the years

The performance of the group is in general equal to or better in comparison to the performance in previous years, except Copper and Strontium. The performance is in general also in line with the precision requirements of EN71-3:19 table C.1.

4.4 EVALUATION OF ANALYTICAL DETAILS

Almost all of the reporting participants mentioned that they are ISO/IEC17025 accredited for the category 1 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 the following can be summarized:

- All, but one participant mentioned to have used a sample intake of 100 mg or more. Please note that test method EN71-3 mentions to take not less than 100 mg whenever possible.
- Almost all participants mentioned to have used a volume ratio of 5 mL of HCl solution per 100 mg sample intake for the migration.
- 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.

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

5 DISCUSSION

Sample #22550 was used before in proficiency test iis13V02 as sample #13042. In the next table a comparison is given between the two proficiency tests. It is observed that the averages and reproducibility of sample #22550 are in line with the previous PT.

Element	unit	sample #22550			sample #13042		
		n	average	R(calc)	n	average	R(calc)
Copper	mg/kg	27	2507	1610	78	2420	1480
Lead	mg/kg	30	137	49	109	124	77
Zinc	mg/kg	26	6092	2487	76	6128	2330

Table 4: comparison of sample #22550 with sample #13042

When the results of this interlaboratory study are compared to the Migration limits from toy materials for category I 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 components. All reporting laboratories would have rejected sample #22550 for too high level of Copper and Lead.

Element	Category I mg/kg
Aluminum	2250
Antimony	45
Arsenic	3.8
Barium	1500
Boron	1200
Cadmium	1.3
Chromium (III)	37.5
Chromium (VI)	0.02
Cobalt	10.5
Copper	622.5
Lead	2.0
Manganese	1200
Mercury	7.5
Nickel	75
Selenium	37.5
Strontium	4500
Tin	15000
Organic Tin	0.9
Zinc	3750

Table 5: Migration limits from toy materials for Category I as mentioned in EN71-3:19+A1:21

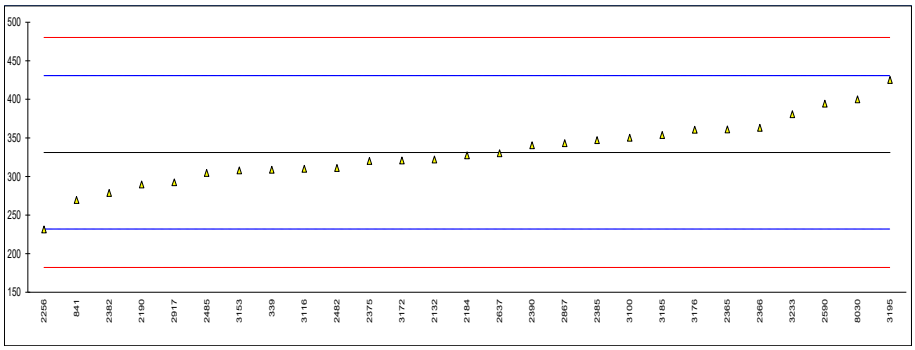
6 CONCLUSION

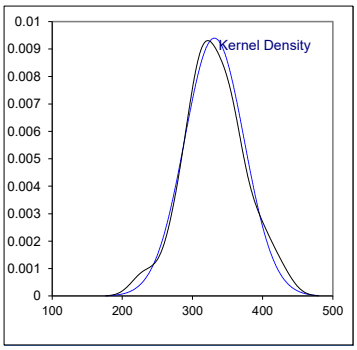
In this PT it appeared that version of EN71-3:19+A1:21 has been followed well by most of the participants. Most of the participants had detected the elements correctly in the samples. Each participating laboratory should evaluate its performance in this study and decide about any corrective actions if 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 plaster sample #22550; results in mg/kg

lab	method	value	mark	z(targ)	remarks
310		-----		-----	
339	EN71-3	308.573		-0.45	
551		-----		-----	
841	EN71-3	269.4		-1.24	
2132	EN71-3	322.0		-0.18	
2184	EN71-3	327.2		-0.08	
2190	EN71-3	289.64		-0.84	
2256	EN71-3	231.22		-2.01	
2301		-----		-----	
2365	EN71-3	361.0		0.60	
2366	EN71-3	363.0		0.64	
2375	EN71-3	320		-0.22	
2382	EN71-3	278.7		-1.06	
2385	EN71-3	347		0.32	
2390	EN71-3	340.284	C	0.18	first reported: 477.5
2482	EN71-3	311		-0.41	
2485	EN71-3	304.419		-0.54	
2590	EN71-3	394.404		1.27	
2637	EN71-3	330		-0.02	
2864		-----		-----	
2867	EN71-3	343.1		0.24	
2917	EN71-3	292.27		-0.78	
3100	EN71-3	350.183		0.38	
3116	EN71-3	309.855		-0.43	
3146		-----		-----	
3153	EN71-3	307.75		-0.47	
3172	EN71-3	320.61	C	-0.21	first reported: 202
3176	EN71-3	360.51		0.59	
3185	EN71-3	353.64		0.45	
3195	EN71-3	425		1.89	
3233	EN71-3	380.49		0.99	
8005		-----		-----	
8030	EN71-3	399.91		1.38	
	normality	OK			
	n	27			
	outliers	0			
	mean (n)	331.154			
	st.dev. (n)	42.4429	RSD = 13%		
	R(calc.)	118.840			
	st.dev.(EN71-3:19+A1:21)	49.6731			
	R(EN71-3:19+A1:21)	139.085			

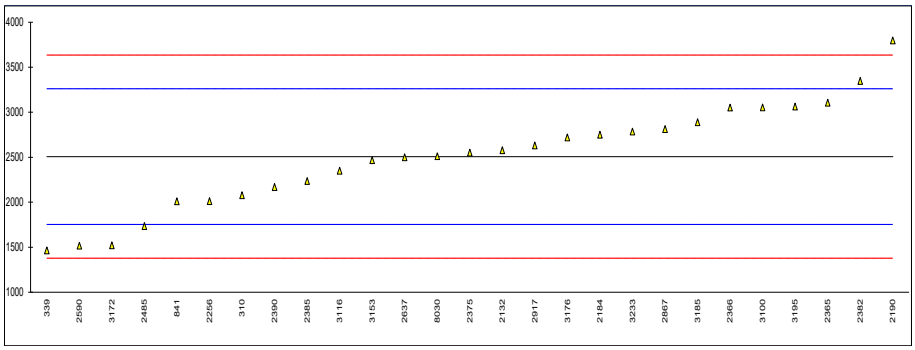


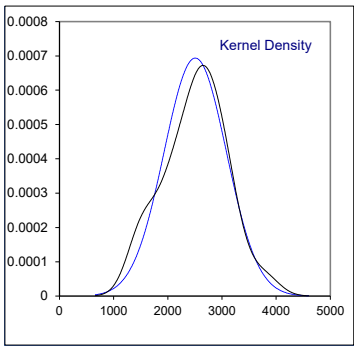


Determination of migration of Copper as Cu on plaster sample #22550; results in mg/kg

lab	method	value	mark	z(targ)	remarks
310	EN71-3	2076.106		-1.15	
339	EN71-3	1464.497	C	-2.77	first reported: 414.593
551		-----		-----	
841	EN71-3	2009.0		-1.32	
2132	EN71-3	2578.2		0.19	
2184	EN71-3	2750		0.65	
2190	EN71-3	3795.49		3.43	
2256	EN71-3	2010.90		-1.32	
2301		-----		-----	
2365	EN71-3	3105.0		1.59	
2366	EN71-3	3050.2		1.45	
2375	EN71-3	2550		0.12	
2382	EN71-3	3345.5		2.23	
2385	EN71-3	2235		-0.72	
2390	EN71-3	2168		-0.90	
2482		-----		-----	
2485	EN71-3	1734.83		-2.05	
2590	EN71-3	1516.824		-2.63	
2637	EN71-3	2500		-0.02	
2864		-----		-----	
2867	EN71-3	2812.2		0.81	
2917	EN71-3	2630.24		0.33	
3100	EN71-3	3051.699		1.45	
3116	EN71-3	2349.022		-0.42	
3146		-----		-----	
3153	EN71-3	2466.51		-0.11	
3172	EN71-3	1520.7	C	-2.62	first reported: 1325
3176	EN71-3	2718	C	0.56	first reported: 27.8
3185	EN71-3	2888.10		1.01	
3195	EN71-3	3061		1.47	
3233	EN71-3	2783.62		0.74	
8005		-----		-----	
8030	EN71-3	2509.40		0.01	

	normality	OK
	n	27
	outliers	0
	mean (n)	2506.668
	st.dev. (n)	574.8508 RSD = 23%
	R(calc.)	1609.582
	st.dev.(EN71-3:19+A1:21)	376.0002
	R(EN71-3:19+A1:21)	1052.801

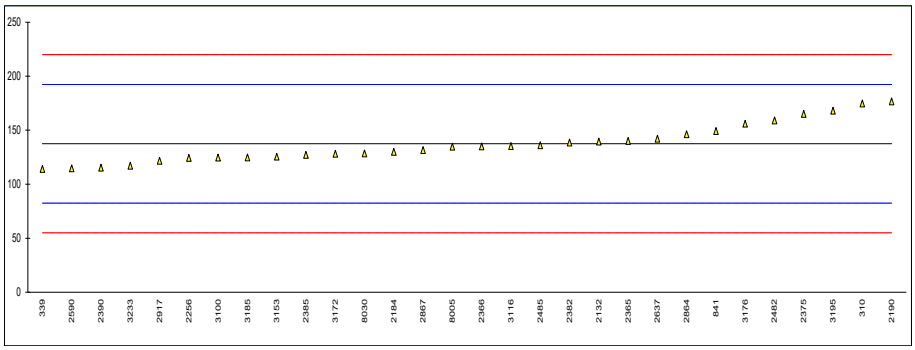


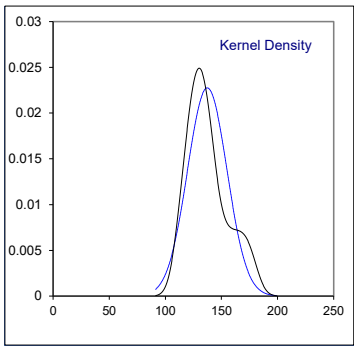


Determination of migration of Lead as Pb on plaster sample #22550; results in mg/kg

lab	method	value	mark	z(targ)	remarks
310	EN71-3	174.626		1.35	
339	EN71-3	114.103	C	-0.85	first reported: 26.025
551		-----		-----	
841	EN71-3	149.2		0.43	
2132	EN71-3	139.4		0.07	
2184	EN71-3	130.0		-0.27	
2190	EN71-3	176.67		1.43	
2256	EN71-3	124.30		-0.48	
2301		-----		-----	
2365	EN71-3	140.0		0.09	
2366	EN71-3	134.9		-0.09	
2375	EN71-3	165		1.00	
2382	EN71-3	138.6	C	0.04	first reported: 176.7
2385	EN71-3	127		-0.38	
2390	EN71-3	115.25		-0.81	
2482	EN71-3	159		0.79	
2485	EN71-3	135.931		-0.05	
2590	EN71-3	114.696		-0.83	
2637	EN71-3	142		0.17	
2864	EN71-3	146.28		0.32	
2867	EN71-3	131.6		-0.21	
2917	EN71-3	121.67		-0.57	
3100	EN71-3	124.605		-0.47	
3116	EN71-3	135.258		-0.08	
3146		-----		-----	
3153	EN71-3	125.40		-0.44	
3172	EN71-3	128.16	C	-0.34	first reported: 74.50
3176	EN71-3	156	C	0.68	first reported: 204.92
3185	EN71-3	124.87		-0.46	
3195	EN71-3	168		1.11	
3233	EN71-3	117.07		-0.74	
8005	F963/GB6675/ISO8124-3	134.685		-0.10	
8030	EN71-3	128.43		-0.33	

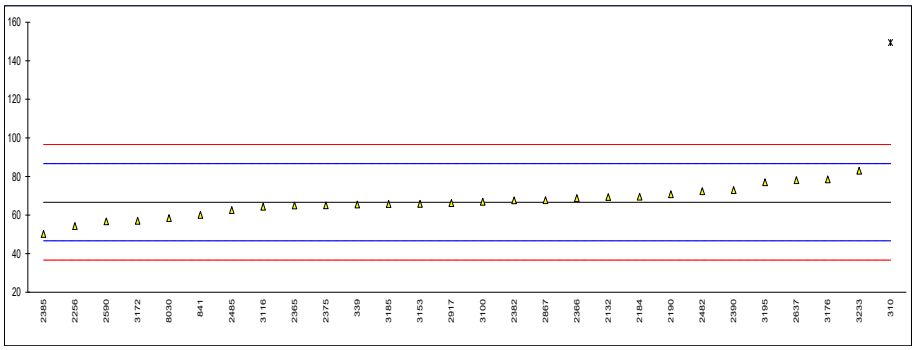
	normality	OK
	n	30
	outliers	0
	mean (n)	137.423
	st.dev. (n)	17.5368 RSD = 13%
	R(calc.)	49.103
	st.dev.(EN71-3:19+A1:21)	27.4847
	R(EN71-3:19+A1:21)	76.957

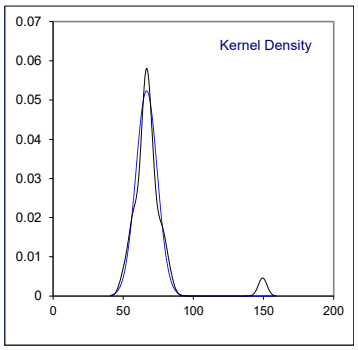




Determination of migration of Manganese as Mn on plaster sample #22550; results in mg/kg

lab	method	value	mark	z(targ)	remarks
310	EN71-3	149.368	R(0.01)	8.28	
339	EN71-3	65.399		-0.12	
551		-----		-----	
841	EN71-3	60.1		-0.65	
2132	EN71-3	69.3		0.27	
2184	EN71-3	69.53		0.29	
2190	EN71-3	70.76		0.41	
2256	EN71-3	54.30		-1.23	
2301		-----		-----	
2365	EN71-3	64.90		-0.17	
2366	EN71-3	68.8		0.22	
2375	EN71-3	65		-0.16	
2382	EN71-3	67.6		0.10	
2385	EN71-3	50.2		-1.64	
2390	EN71-3	73		0.64	
2482	EN71-3	72.4		0.58	
2485	EN71-3	62.561		-0.41	
2590	EN71-3	56.710		-0.99	
2637	EN71-3	78		1.14	
2864		-----		-----	
2867	EN71-3	67.71		0.11	
2917	EN71-3	66.19		-0.04	
3100	EN71-3	66.852		0.02	
3116	EN71-3	64.372		-0.23	
3146		-----		-----	
3153	EN71-3	65.80		-0.08	
3172	EN71-3	57.01	C	-0.96	first reported: <50
3176	EN71-3	78.49		1.19	
3185	EN71-3	65.68		-0.10	
3195	EN71-3	77.0		1.04	
3233	EN71-3	82.94		1.63	
8005		-----		-----	
8030	EN71-3	58.42		-0.82	
	normality	OK			
	n	27			
	outliers	1			
	mean (n)	66.631			
	st.dev. (n)	7.6218	RSD = 11%		
	R(calc.)	21.341			
	st.dev.(EN71-3:19+A1:21)	9.9946			
	R(EN71-3:19+A1:21)	27.985			

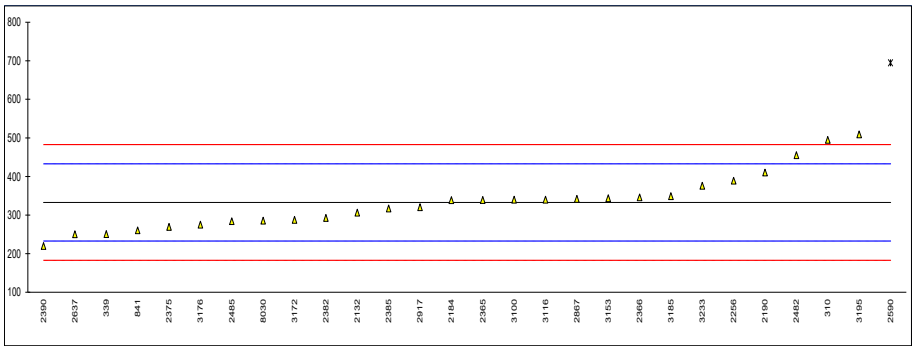


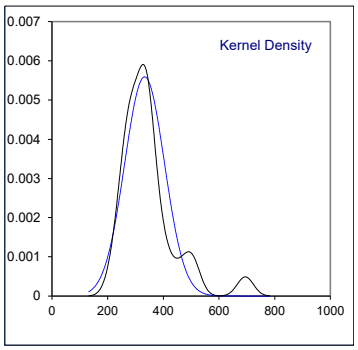


Determination of migration of Strontium as Sr on plaster sample #22550; results in mg/kg

lab	method	value	mark	z(targ)	remarks
310	EN71-3	494.516		3.24	
339	EN71-3	250.561		-1.65	
551		-----		-----	
841	EN71-3	260.2		-1.46	
2132	EN71-3	305.8		-0.54	
2184	EN71-3	338.6		0.11	
2190	EN71-3	410.08		1.55	
2256	EN71-3	388.74		1.12	
2301		-----		-----	
2365	EN71-3	339.0		0.12	
2366	EN71-3	345.9		0.26	
2375	EN71-3	269		-1.28	
2382	EN71-3	292.4		-0.81	
2385	EN71-3	317		-0.32	
2390	EN71-3	219.70		-2.27	
2482	EN71-3	455		2.45	
2485	EN71-3	284.139		-0.98	
2590	EN71-3	694.446	C,R(0.01)	7.24	first reported: 729.367
2637	EN71-3	250		-1.66	
2864		-----		-----	
2867	EN71-3	341.8		0.18	
2917	EN71-3	320.32		-0.25	
3100	EN71-3	339.688		0.14	
3116	EN71-3	339.988		0.14	
3146		-----		-----	
3153	EN71-3	343.61		0.22	
3172	EN71-3	287.55	C	-0.91	first reported: 187.5
3176	EN71-3	275	C	-1.16	first reported: 1282.3
3185	EN71-3	348.55		0.31	
3195	EN71-3	509		3.53	
3233	EN71-3	375.70		0.86	
8005		-----		-----	
8030	EN71-3	285.40		-0.95	

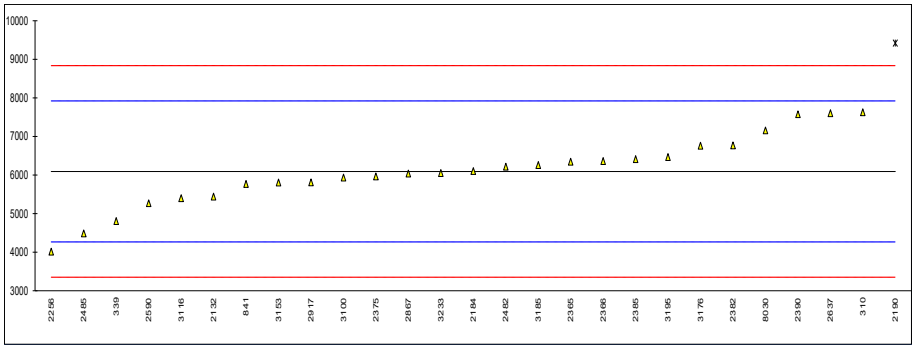
	normality	OK
	n	27
	outliers	1
	mean (n)	332.861
	st.dev. (n)	71.3559 RSD = 21%
	R(calc.)	199.797
	st.dev.(EN71-3:19+A1:21)	49.9291
	R(EN71-3:19+A1:21)	139.802

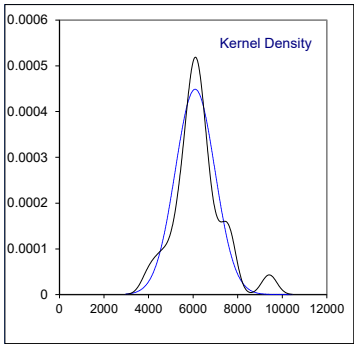




Determination of migration of Zinc as Zn on plaster sample #22550; results in mg/kg

lab	method	value	mark	z(targ)	remarks
310	EN71-3	7624.467		1.68	
339	EN71-3	4806.449	C	-1.41	first reported: 3333.184
551		-----		-----	
841	EN71-3	5765.6		-0.36	
2132	EN71-3	5440.8		-0.71	
2184	EN71-3	6100		0.01	
2190	EN71-3	9418.17	R(0.05)	3.64	
2256	EN71-3	4009.7	C	-2.28	first reported: 3674.57
2301		-----		-----	
2365	EN71-3	6340.0		0.27	
2366	EN71-3	6357.2		0.29	
2375	EN71-3	5960		-0.14	
2382	EN71-3	6765.3		0.74	
2385	EN71-3	6410		0.35	
2390	EN71-3	7575		1.62	
2482	EN71-3	6215		0.13	
2485	EN71-3	4484.48		-1.76	
2590	EN71-3	5267.229		-0.90	
2637	EN71-3	7600		1.65	
2864		-----		-----	
2867	EN71-3	6036.1		-0.06	
2917	EN71-3	5810.05		-0.31	
3100	EN71-3	5932.330		-0.17	
3116	EN71-3	5400.595		-0.76	
3146		-----		-----	
3153	EN71-3	5803.03		-0.32	
3172		-----		-----	
3176	EN71-3	6755.0		0.73	
3185	EN71-3	6259.54		0.18	
3195	EN71-3	6465		0.41	
3233	EN71-3	6051.86		-0.04	
8005		-----		-----	
8030	EN71-3	7154.34		1.16	
	normality	OK			
	n	26			
	outliers	1			
	mean (n)	6091.888			
	st.dev. (n)	888.3896	RSD = 15%		
	R(calc.)	2487.491			
	st.dev.(EN71-3:19+A1:21)	913.7832			
	R(EN71-3:19+A1:21)	2558.593			





APPENDIX 2

Determination of migration of other elements on plaster sample #22550; results in mg/kg

lab	Sb	As	Ba	B	Cd	Cr (III)	Cr (VI)
310	0.148	0.499	5.898	10.024	0.135	<1.0	<0.015
339	0.134	0.316	3.716	7.698	0.174	0.525	-----
551	-----	-----	-----	-----	-----	-----	-----
841	<0.1	0.35	3.2	4.3	0.15	0.288	<0.005
2132	<2.5	0.494	<25	<25	<0.25	<1	<0.005
2184	not detected	not detected	6.71	not detected	0.19	not detected	not detected
2190	<10	<0.5	<50	<50	0.18	<5	nd
2256	not detected	0.54	5.90	4.80	0.14	0.29	not detected
2301	-----	-----	-----	-----	-----	-----	-----
2365	<1	<0.5	<50	<50	<0.1	<5	<0.010
2366	<1	<0.5	<50	<50	<0.1	<1	<0.01
2375	<1	<0.5	<50	<50	<0.1	<5	<0.01
2382	＜1	＜0.5	＜50	＜50	0.1485	＜5	＜0.01
2385	<1	<1	3.57	7.90	<0.5	<0.5	<0.02
2390	0.75	Not detected	Not detected	11.20	0.15	0.250	not analyzed
2482	-----	-----	-----	-----	0.150	0.399	-----
2485	-----	0.293	4.818	6.361	0.136	-----	-----
2590	< L.O.Q.	< L.O.Q.	10.863	-----	< L.O.Q.	< L.O.Q.	-----
2637	<0,1	0.5	5.5	6	0.17	0.3	<0,1
2864	not detected	not detected	<10	-----	not detected	-----	-----
2867	<1	<0.5	<10	<10	<0.5	<1	<0.015
2917	<0.25	<0.5	4.42	6.40	<0.5	<0.5	-----
3100	<10	<1.0	<10	<50	<0.5	<5	<0.01
3116	<2	0.428	7.445	7.693	<0.15	<1	<0.002
3146	-----	-----	-----	-----	-----	-----	-----
3153	<10	<1	<10	<50	<0.5	<5	<0.01
3172	< 10	< 1	< 50	< 50	0.1215	< 10	< 0.005
3176	0.57	0.50	4.2	5.0	0.20	-----	-----
3185	<10	<1	<10	<50	<0.5	<5	<0.01
3195	<0,50	0.510	7.83	8.34	0.186	-----	-----
3233	< 5	0.47	6.79	7.21	0.14	0.47	< 0.0025
8005	<2	<2	7.411	-----	<2	-----	-----
8030	Not detected	Not detected	5.90	Not detected	Not detected	0.31	Not detected

Determination of migration of other elements on plaster sample #22550; results in mg/kg
--continued--

lab	Co	Hg	Ni	Se	Sn	Org.Sn
310	0.327	<0.5	0.709	0.676	<0.25	-----
339	0.236	0	0.677	0.448	0	-----
551	-----	-----	-----	-----	-----	-----
841	0.2	<0.1	<2.5	<0.5	<0.025	<0.04
2132	0.270	<0.25	<2.5	<2.5	<15	N/A
2184	not detected	not detected	not detected	not detected	not detected	not detected
2190	<1	<1	<10	<5	<4	<0.25
2256	not detected	not detected	0.79	not detected	not detected	not detected
2301	-----	-----	-----	-----	-----	-----
2365	<0.5	<0.5	<10	<5	<0.36	-----
2366	<0.5	<0.5	<10	<5	<0.15	<0.15
2375	<0.5	<0.5	<10	<5	<0.36	-----
2382	＜0.5	＜0.5	＜10	＜5	＜0.150	＜0.02
2385	<0.5	<0.1	<1	<1	<1	<0.2
2390	Not detected	Not detected	Not detected	Not detected	Not detected	Not analyzed
2482	-----	-----	-----	-----	-----	-----
2485	0.234	-----	-----	0.361	-----	-----
2590	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	-----
2637	0.3	<0,1	1	0.8	<0,5	-----
2864	-----	not detected	-----	not detected	-----	-----
2867	<0.5	<0.5	<2.5	<2.5	<0.2	<0.2
2917	<0.25	<0.1	<1	<0.5	<0.25	-----
3100	<1.0	<1.0	<10	<5	<0.1	<0.3
3116	<2	<0.15	<2	<2	<2	<0.225
3146	-----	-----	-----	-----	-----	not detected
3153	<1	<1	<10	<5	<10	<0.3
3172	< 5	< 5	< 10	< 10	< 50	< 0.05
3176	0.31	-----	0.29	0.63	-----	-----
3185	<1	<1	<10	<5	<0.1	<0.3
3195	0.296	<0,10	<2,0	0.824	<1,0	-----
3233	< 0.5	< 0.5	< 5	< 5	0.19	< 0.04
8005	-----	<2	-----	<2	-----	-----
8030	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected

APPENDIX 3 Analytical details

lab	ISO/IEC17025 accredited	Sample intake	Amount 0.07 mol/L HCl solution used (mL)	pH after shaking	pH adjusted after shaking	pH after adjustment
310	Yes	0.2757	14	1.6	Yes	1.2
339	Yes	0.256 g	12.8 mL	1.37	Yes	1.27
551	---				---	
841	Yes	0.2 grams	10 ml	<1.3	Yes	<1.3
2132	Yes	0.1003 gram	5 mL	1.62	Yes	1.20
2184	Yes	0.1g	5ml	1.46	Yes	1.20
2190	Yes	0.2g	10mL	1.16	no	
2256	Yes	0.1858 gram	9.3	1.217	No	N/A
2301	---				---	
2365	Yes	0.1g	5mL	1.50	Yes	1.18
2366	Yes	0.15	7.5	1.46	Yes	1.15
2375	Yes				---	
2382	Yes	0.2g	10ml	1.40	Yes	1.27
2385	Yes	~ 0.5 g	25 mL	1.434	Yes	1.281
2390	Yes	0.2060	10.3ml	1.2	No	
2482	Yes	0.2g	10	1.4	Yes	1.24
2485	Yes	0.1 grams	5 mL	1.29	No	
2590	Yes	0.1	10ML	1.71	Yes	1.12
2637	Yes	0,4 g	20 ml		---	
2864	Yes	100 mg	5 mL	1.7	Yes	1.3
2867	Yes	0.2g	10ml	1.2	No	NA
2917	Yes	0.2574 and 0.2508	15.1 and 15.1	1.4 and 1.6	Yes	1.2 and 1.2
3100	Yes	0.1g	5mL	1.42	Yes	1.12
3116	Yes	0.25g	12.5	Between 1.1-1.2	No	Not applicable
3146	No	0,544 g	27,4 mL	1,3	No	
3153	Yes	0.1 gram	5 mL	1.42	Yes	1.19
3172	Yes				---	
3176	Yes	0,05	50	1,25	No	
3185	Yes	0.12g	6mL	1.47	Yes	1.18
3195	No	0,2g	10mL	2,5	Yes	1,2
3233	Yes	0.1118 g	5.6 mL	1.36	Yes	1.16
8005	Yes	0.25g	12.5 mL	1.58	Yes	1.1-1.2
8030	Yes	0.4990g/Organic Tin 0.5g	25 mL	2.18	Yes	1.10

APPENDIX 4

Number of participants per country

1 lab in BRAZIL
3 labs in FRANCE
5 labs in GERMANY
5 labs in HONG KONG
1 lab in INDONESIA
2 labs in ITALY
7 labs in P.R. of CHINA
1 lab in PAKISTAN
1 lab in SLOVENIA
1 lab in SWITZERLAND
1 lab in TAIWAN
1 lab in THAILAND
1 lab in THE NETHERLANDS
2 labs in TURKEY
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
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

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
5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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)