Institute for
Interlaboratory Studies

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

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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 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 Migration of elements. This proficiency test describes the Migration of elements EN71-3 for category I samples.

In the interlaboratory study 43 laboratories in 17 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 I 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 #23555.
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 artificially fortified with some elements was selected. After homogenization 50 plastic bags were filled with approximately 0.5 grams each and labelled #23555. The batch for sample #23555 was used in a previous proficiency test on Migration of elements as sample #17555 in iis17V02. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one plaster sample of 0.5 grams labelled #23555 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 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:

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

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 no problems were encountered with the dispatch of the samples. Two participants reported test results after the final reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.
In total 40 reporting laboratories reported 226 numerical test results. Observed were 7 outlying test results, which is 3.1%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER 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. Therefore, based on the answers given by the participants, some of test results are excluded from the statistical evaluation when the mentioned pH values are outside the range of 1.1 and 1.3.

Aluminum as Al: This determination was not problematic. Four statistical outliers were observed and two other test results were excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN71-3:19+A1:21.
No effect is observed when evaluated over test results based on a pH between 1.1-1.3 only.

Antimony as Sb: This determination was problematic. No statistical outliers were observed but two test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN71-3:19+A1:21.
No effect is observed when evaluated over test results based on a pH between 1.1-1.3 only.

Copper as Cu: This determination was problematic. No statistical outliers were observed but two test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN71-3:19+A1:21.
No effect is observed when evaluated over test results based on a pH between 1.1-1.3 only.

Lead as Pb: This determination was not problematic. No statistical outliers were observed but three test results were excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN71-3:19+A1:21.
No effect is observed when evaluated over test results based on a pH between 1.1-1.3 only.

Manganese as Mn: This determination was not problematic. Two statistical outliers were observed and two other test results were excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN71-3:19+A1:21.
No effect is observed when evaluated over test results based on a pH between 1.1-1.3 only.

Strontium as Sr: This determination was not problematic. One statistical outlier was observed and two other test results were excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN71-3:19+A1:21.
No effect is observed when evaluated over test results based on a pH between 1.1-1.3 only.

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

Element	unit	n	average	2.8 * sd	R(lit)
Aluminum as Al	mg/kg	31	338	86	142
Antimony as Sb	mg/kg	37	17.8	19.2	14.9
Copper as Cu	mg/kg	35	56.7	45.7	23.8
Lead as Pb	mg/kg	37	40.6	19.7	22.7
Manganese as Mn	mg/kg	33	68.2	23.9	28.6
Strontium as Sr	mg/kg	33	343	123	144

Table 1: reproducibilities of tests on sample #23555

Without further statistical calculations it can be concluded that for most 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 2023 WITH PREVIOUS PTS

	April 2023	April 2022	April 2021	April 2020	April 2019
Number of reporting laboratories	40	31	38	37	36
Number of test results	226	167	197	243	123
Number of statistical outliers	7	3	11	4	8
Percentage of statistical outliers	3.1%	1.8%	5.6%	1.6%	6.5%

Table 2: 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	2019-2013	Target
Aluminum	9%	13%	34%	17%	20-29%	15%
Antimony	39%	---	15%	---	28-47%	30%
Arsenic	---	---	13%	---	32%	20%
Barium	---	---	---	---	20%	20%
Boron	---	---	---	---	---	15%
Cadmium	---	---	14%	14%	18%	20%

Element	April 2023	April 2022	April 2021	April 2020	2019-2013	Target
Chromium (III)	---	---	---	---	44%	15%
Chromium (VI)	---	---	---	---	---	50%
Cobalt	---	---	---	---	---	15%
Copper	29%	23%	---	---	10-28%	15%
Lead	17%	13%	17%	23%	18-30%	20%
Manganese	13%	11%	---	10%	12-16%	15%
Mercury	---	---	---	---	---	30%
Nickel	---	---	10%	---	15%	20%
Selenium	---	---	---	---	25%	15%
Strontium	13%	21%	10%	12%	15-21%	15%
Tin	---	---	---	---	---	20%
Organic Tin	---	---	---	---	---	50%
Zinc	---	15%	---	12%	14-17%	15%

Table 3: development of uncertainties over the years

The uncertainties observed in this PT are comparable to the uncertainties observed in previous PTs, except Antimony and Copper The uncertainty is large in comparison with the requirements mentioned in the target for Antimony and Copper.

Sample #23555 was used before in proficiency test iis17V02 as sample #17555. In the next table a comparison is given between the two proficiency tests. The findings in current PT are in line with the previous PT (see table 4).

Element	unit	sample #23555			sample #17555		
		n	average	R(calc)	n	average	R(calc)
Aluminum	mg/kg	31	338	86	73	316	204
Antimony	mg/kg	37	17.8	19.2	73	25.5	33.6
Copper	mg/kg	35	56.7	45.7	70	56.2	44.7
Lead	mg/kg	37	40.6	19.7	78	38.5	24.1
Manganese	mg/kg	33	68.2	23.9	70	65.0	23.7
Strontium	mg/kg	33	343	123	73	329	191

Table 4: comparison of sample #23555 with sample #17555

4.4 EVALUATION OF ANALYTICAL DETAILS

Almost all reporting participants mentioned that they are ISO/IEC17025 accredited for the category I 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:

- About 47% of the participants have used a sample intake around 100 mg, about 34% have used a sample intake between 200 - 300 mg and about 16% a sample intake between 400 – 500 mg. Please note that test method EN71-3 mentions to take not less than 100 mg whenever possible.

- About 89% of the participants mentioned to have used a volume ratio of 5 mL of HCl solution per 100 mg sample intake for the migration.
- Most participants (75%) have used a solution with a pH between 1.1 and 1.3 (with or without adjustment) for the determination of the elements. Three participants did not keep the pH correctly, see for more details paragraph 4.1. And seven participants did not mention how the pH was controlled.

As the majority of the group follow almost the same analytical procedures no separate statistical analysis has been performed except the effect of the pH. This evaluation is mentioned in paragraph 4.1 and appendix 1.

5 DISCUSSION

It appeared that EN71-3:19+A1:21 has been followed by most of the participants, except for a small group that did not follow the adjustment of the pH and the use of the volume ratio correctly. The effect of the pH on the determination seems neglectable.

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 participants would have rejected sample #23555 for too high level of 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

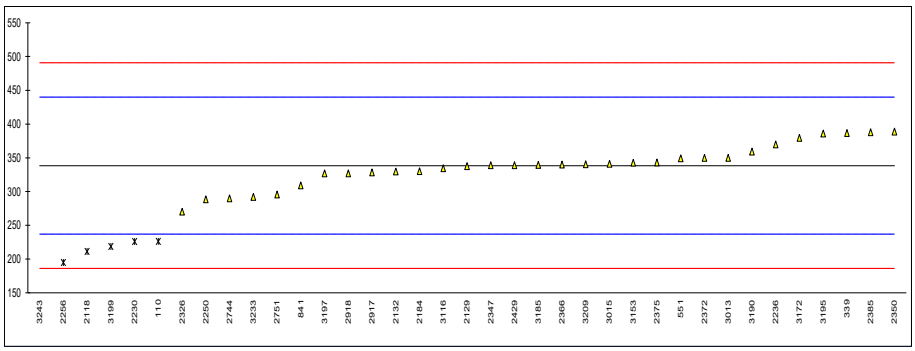
6 CONCLUSION

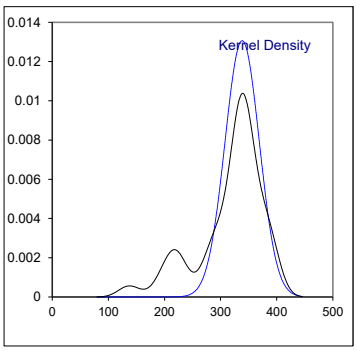
In this PT it appeared that version of EN71-3:19+A1:21 has been followed mostly well by the participants. Most of the participants had detected the elements correctly in the samples. Each participant should evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, 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 #23555; results in mg/kg

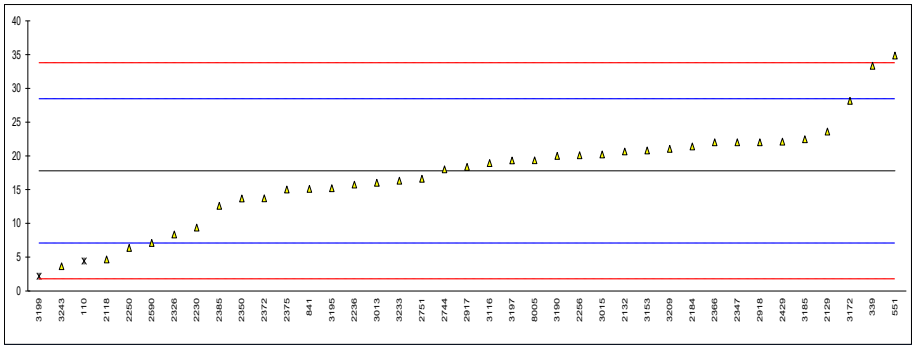
lab	method	value	mark	z(targ)	remarks
110	EN71-3	226.109	ex	-2.21	test result excluded, see § 4.1
339	EN71-3	386.662		0.95	
551	EN71-3	349.16		0.21	
841	EN71-3	309.1		-0.58	
2118	EN71-3	211.13	R(0.05)	-2.51	
2129	EN71-3	337.563		-0.02	
2132	EN71-3	329.61		-0.17	
2184	EN71-3	330.2		-0.16	
2230	EN71-3	226	C,R(0.05)	-2.21	first reported 222
2236	In house	369.8		0.62	
2250	EN71-3	288.5		-0.98	
2256	EN71-3	194.90	R(0.05)	-2.83	
2326	EN71-3	270.12		-1.35	
2347	EN71-3	339		0.01	
2350	EN71-3	388.95		1.00	
2366	EN71-3	340		0.03	
2372	EN71-3	350		0.23	
2375	EN71-3	343		0.09	
2381		-----		-----	
2385	EN71-3	388		0.98	
2429	EN71-3	339.1		0.01	
2495		-----		-----	
2590		-----		-----	
2744	EN71-3	290		-0.95	
2751	EN71-3	295.50		-0.85	
2917	EN71-3	328.29		-0.20	
2918	EN71-3	327		-0.23	
2984		-----		-----	
3013	EN71-3	350		0.23	
3015	EN71-3	341		0.05	
3116		334.35		-0.08	
3124		-----		-----	
3153	EN71-3	342.66		0.08	
3172	EN71-3	379.3		0.81	
3185	EN71-3	339.43		0.02	
3190	EN71-3	359		0.41	
3195	EN71-3	386		0.94	
3197	EN71-3	326.97		-0.23	
3199	EN71-3	218.58	ex	-2.36	test result excluded, see § 4.1
3209	EN71-3	340.61		0.04	
3233	EN71-3	292.24		-0.91	
3243	EN71-3	137.245	R(0.05)	-3.96	
8005		-----		-----	
					Only test results that maintain pH between 1.1 – 1,3
	normality	OK			OK
	n	31			26
	outliers	4 + 2ex			3
	mean (n)	338.423			338.422
	st.dev. (n)	30.5411	RSD = 9%		30.7677 RSD = 9%
	R(calc.)	85.515			86.150
	st.dev.(EN71-3:19+A1:21)	50.7635			50.7633
	R(EN71-3:19+A1:21)	142.138			142.137

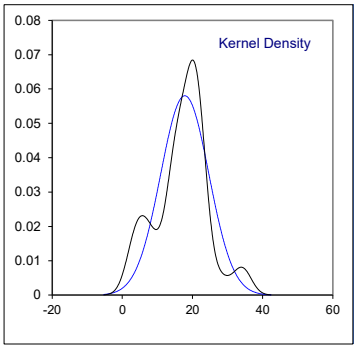




Determination of migration of Antimony as Sb on plaster sample #23555; results in mg/kg

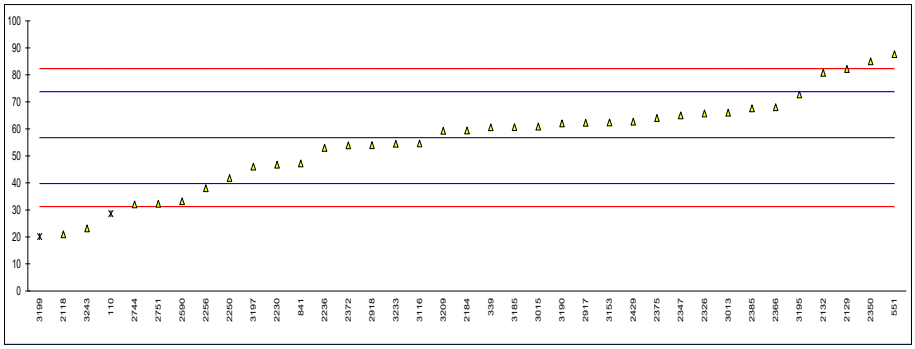
lab	method	value	mark	z(targ)	remarks
110	EN71-3	4.420	ex	-2.50	test result excluded, see § 4.1
339	EN71-3	33.344		2.92	
551	EN71-3	34.867		3.20	
841	EN71-3	15.1		-0.50	
2118	EN71-3	4.66		-2.46	
2129	EN71-3	23.591		1.09	
2132	EN71-3	20.65		0.54	
2184	EN71-3	21.39		0.68	
2230	EN71-3	9.38		-1.58	
2236	In house	15.73		-0.39	
2250	EN71-3	6.352		-2.14	
2256	EN71-3	20.09		0.43	
2326	EN71-3	8.37		-1.76	
2347	EN71-3	22		0.79	
2350	EN71-3	13.68		-0.77	
2366	EN71-3	22		0.79	
2372	EN71-3	13.7		-0.77	
2375	EN71-3	15		-0.52	
2381		-----		-----	
2385	EN71-3	12.6		-0.97	
2429	EN71-3	22.1		0.81	
2495		-----		-----	
2590	EN71-3	7.11		-2.00	
2744	EN71-3	18	C	0.04	first reported 35
2751	EN71-3	16.61	C	-0.22	first reported 32.85
2917	EN71-3	18.34		0.10	
2918	EN71-3	22.0		0.79	
2984	ISO8124-3	not detected		-----	possibly a false negative test result?
3013	EN71-3	16		-0.33	
3015	EN71-3	20.2		0.45	
3116		18.92		0.21	
3124		-----		-----	
3153	EN71-3	20.78		0.56	
3172	EN71-3	28.14		1.94	
3185	EN71-3	22.47		0.88	
3190	EN71-3	20		0.42	
3195	EN71-3	15.2		-0.48	
3197	EN71-3	19.31		0.29	
3199	EN71-3	2.18	ex	-2.92	test result excluded, see § 4.1
3209	EN71-3	21.02		0.61	
3233	EN71-3	16.31		-0.28	
3243	EN71-3	3.648		-2.65	
8005	ASTM F963	19.35		0.29	
					Only test results that maintain pH between 1.1 – 1,3
	normality	OK			suspect
	n	37			30
	outliers	0 + 2ex			0
	mean (n)	17.784			18.120
	st.dev. (n)	6.8737	RSD = 39%		6.5259 RSD = 36%
	R(calc.)	19.246			18.273
	st.dev.(EN71-3:19+A1:21)	5.3352			5.4360
	R(EN71-3:19+A1:21)	14.939			15.221

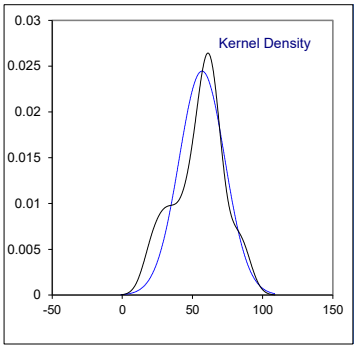




Determination of migration of Copper as Cu on plaster sample #23555; results in mg/kg

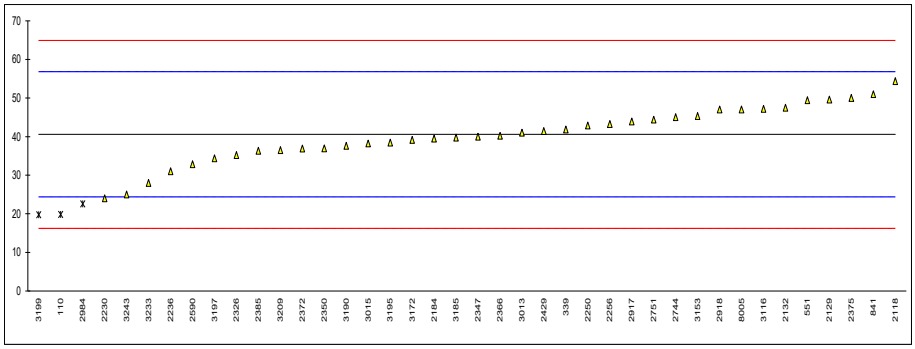
lab	method	value	mark	z(targ)	remarks
110	EN71-3	28.596	ex	-3.31	test result excluded, see § 4.1
339	EN71-3	60.532		0.45	
551	EN71-3	87.643		3.63	
841	EN71-3	47.2		-1.12	
2118	EN71-3	20.94		-4.21	
2129	EN71-3	82.140		2.99	
2132	EN71-3	80.72		2.82	
2184	EN71-3	59.39		0.31	
2230	EN71-3	46.7		-1.18	
2236	In house	52.94		-0.45	
2250	EN71-3	41.83		-1.75	
2256	EN71-3	38.04		-2.20	
2326	EN71-3	65.68		1.05	
2347	EN71-3	65		0.97	
2350	EN71-3	85		3.32	
2366	EN71-3	68		1.32	
2372	EN71-3	53.9		-0.33	
2375	EN71-3	64		0.85	
2381		-----		-----	
2385	EN71-3	67.6		1.28	
2429	EN71-3	62.7		0.70	
2495		-----		-----	
2590	EN71-3	33.16	C	-2.77	first reported 20.23
2744	EN71-3	32		-2.91	
2751	EN71-3	32.27		-2.87	
2917	EN71-3	62.27		0.65	
2918	EN71-3	54.0		-0.32	
2984		-----		-----	
3013	EN71-3	66		1.09	
3015	EN71-3	60.8		0.48	
3116		54.59		-0.25	
3124		-----		-----	
3153	EN71-3	62.38		0.66	
3172	EN71-3	< 50		-----	
3185	EN71-3	60.63		0.46	
3190	EN71-3	62		0.62	
3195	EN71-3	72.7		1.88	
3197	EN71-3	45.98		-1.26	
3199	EN71-3	20.1	ex	-4.30	test result excluded, see § 4.1
3209	EN71-3	59.28		0.30	
3233	EN71-3	54.44		-0.27	
3243	EN71-3	23.172		-3.94	
8005		-----		-----	
					Only test results that maintain pH between 1.1 – 1,3
	normality	OK			OK
	n	35			30
	outliers	0 + 2ex			0
	mean (n)	56.732			57.734
	st.dev. (n)	16.3057	RSD = 29%		16.0213 RSD = 28%
	R(calc.)	45.656			44.860
	st.dev.(EN71-3:19+A1:21)	8.5098			8.6602
	R(EN71-3:19+A1:21)	23.828			24.248

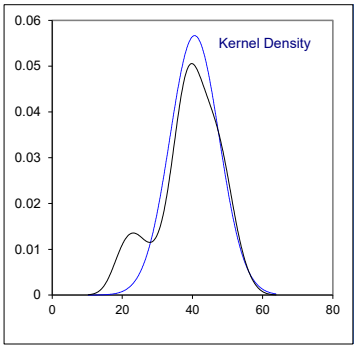




Determination of migration of Lead as Pb on plaster sample #23555; results in mg/kg

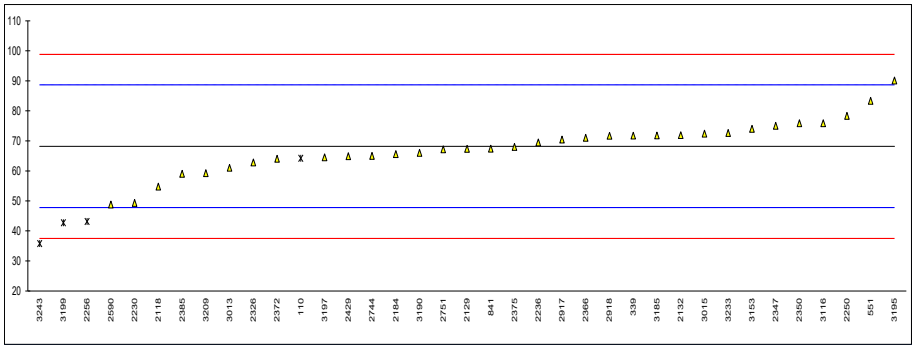
lab	method	value	mark	z(targ)	remarks
110	EN71-3	19.801	ex	-2.56	test result excluded, see § 4.1
339	EN71-3	41.855		0.16	
551	EN71-3	49.435		1.09	
841	EN71-3	50.98		1.28	
2118	EN71-3	54.35		1.70	
2129	EN71-3	49.569		1.11	
2132	EN71-3	47.47		0.85	
2184	EN71-3	39.47		-0.14	
2230	EN71-3	24.0		-2.04	
2236	In house	31.01		-1.18	
2250	EN71-3	42.85		0.28	
2256	EN71-3	43.25		0.33	
2326	EN71-3	35.18		-0.67	
2347	EN71-3	40		-0.07	
2350	EN71-3	36.95		-0.45	
2366	EN71-3	40.2		-0.05	
2372	EN71-3	36.9		-0.45	
2375	EN71-3	50		1.16	
2381		-----		-----	
2385	EN71-3	36.3		-0.53	
2429	EN71-3	41.4		0.10	
2495		-----		-----	
2590	EN71-3	32.84		-0.95	
2744	EN71-3	45		0.54	
2751	EN71-3	44.39		0.47	
2917	EN71-3	43.93		0.41	
2918	EN71-3	47.0		0.79	
2984	ISO8124-3	22.5550	ex	-2.22	test result excluded, see § 4.1
3013	EN71-3	41		0.05	
3015	EN71-3	38.2		-0.29	
3116		47.18		0.81	
3124		-----		-----	
3153	EN71-3	45.33		0.58	
3172	EN71-3	39.12		-0.18	
3185	EN71-3	39.73		-0.11	
3190	EN71-3	37.6		-0.37	
3195	EN71-3	38.4		-0.27	
3197	EN71-3	34.34		-0.77	
3199	EN71-3	19.71	ex	-2.57	test result excluded, see § 4.1
3209	EN71-3	36.48		-0.51	
3233	EN71-3	27.98		-1.55	
3243	EN71-3	24.989		-1.92	
8005	ASTM F963	47.00		0.79	
					Only test results that maintain pH between 1.1 – 1,3
	normality	OK			OK
	n	37			30
	outliers	0 + 3ex			0
	mean (n)	40.586			40.645
	st.dev. (n)	7.0392	RSD = 17%		7.0668 RSD = 17%
	R(calc.)	19.710			19.787
	st.dev.(EN71-3:19+A1:21)	8.1172			8.1289
	R(EN71-3:19+A1:21)	22.728			22.761

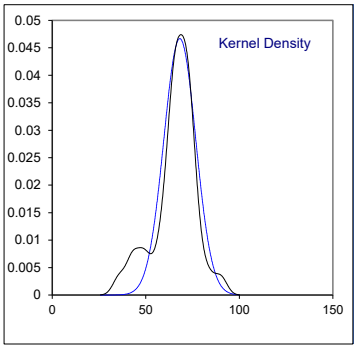




Determination of migration of Manganese as Mn on plaster sample #23555; results in mg/kg

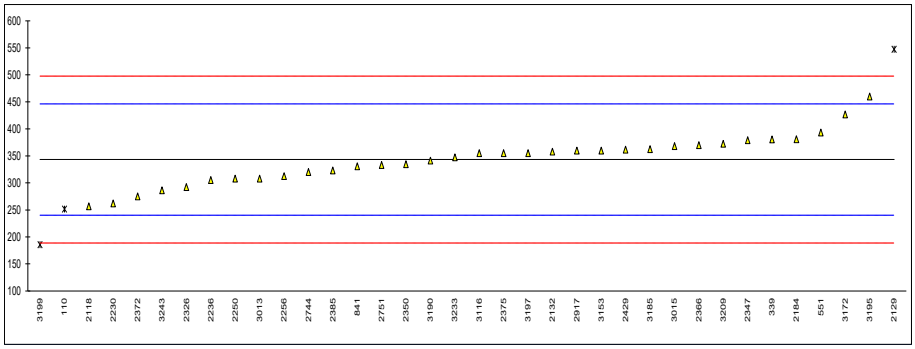
lab	method	value	mark	z(targ)	remarks
110	EN71-3	64.192	ex	-0.39	test result excluded, see § 4.1
339	EN71-3	71.709		0.34	
551	EN71-3	83.307		1.48	
841	EN71-3	67.4		-0.08	
2118	EN71-3	54.75		-1.31	
2129	EN71-3	67.345		-0.08	
2132	EN71-3	71.88		0.36	
2184	EN71-3	65.57		-0.26	
2230	EN71-3	49.3		-1.85	
2236	In house	69.42		0.12	
2250	EN71-3	78.31		0.99	
2256	EN71-3	43.09	DG(0.05)	-2.45	
2326	EN71-3	62.81		-0.53	
2347	EN71-3	75		0.67	
2350	EN71-3	75.86		0.75	
2366	EN71-3	71		0.28	
2372	EN71-3	64		-0.41	
2375	EN71-3	68		-0.02	
2381		-----		-----	
2385	EN71-3	59.0		-0.90	
2429	EN71-3	64.9		-0.32	
2495		-----		-----	
2590	EN71-3	48.76	C	-1.90	first reported 38.14
2744	EN71-3	65		-0.31	
2751	EN71-3	67.15		-0.10	
2917	EN71-3	70.43		0.22	
2918	EN71-3	71.7		0.34	
2984		-----		-----	
3013	EN71-3	61		-0.70	
3015	EN71-3	72.4		0.41	
3116		75.89		0.75	
3124		-----		-----	
3153	EN71-3	74.00		0.57	
3172	EN71-3	< 50		-----	
3185	EN71-3	71.78		0.35	
3190	EN71-3	66		-0.21	
3195	EN71-3	90.1		2.14	
3197	EN71-3	64.51		-0.36	
3199	EN71-3	42.68	ex	-2.49	test result excluded, see § 4.1
3209	EN71-3	59.24		-0.87	
3233	EN71-3	72.58		0.43	
3243	EN71-3	35.781	DG(0.05)	-3.17	
8005		-----		-----	
					Only test results that maintain pH between 1.1 – 1,3
	normality	suspect			suspect
	n	33			29
	outliers	2 + 2ex			1
	mean (n)	68.185			67.721
	st.dev. (n)	8.5482	RSD = 13%		8.6821 RSD = 13%
	R(calc.)	23.935			24.310
	st.dev.(EN71-3:19+A1:21)	10.2277			10.1581
	R(EN71-3:19+A1:21)	28.638			28.443

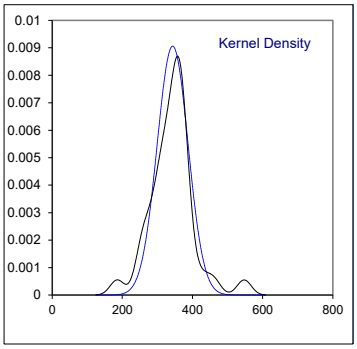




Determination of migration of Strontium as Sr on plaster sample #23555; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	EN71-3	251.440	ex	-1.78	test result excluded, see § 4.1
339	EN71-3	380.291		0.72	
551	EN71-3	393.038		0.97	
841	EN71-3	330.7		-0.25	
2118	EN71-3	256.44		-1.69	
2129	EN71-3	547.237	R(0.01)	3.96	
2132	EN71-3	357.78		0.28	
2184	EN71-3	380.7		0.73	
2230	EN71-3	262	C	-1.58	first reported 209
2236	In house	305.2		-0.74	
2250	EN71-3	307.8		-0.69	
2256	EN71-3	312.52		-0.60	
2326	EN71-3	292.05		-1.00	
2347	EN71-3	379		0.69	
2350	EN71-3	334.56		-0.17	
2366	EN71-3	370		0.52	
2372	EN71-3	275		-1.33	
2375	EN71-3	355		0.23	
2381		-----		-----	
2385	EN71-3	323		-0.39	
2429	EN71-3	361.3		0.35	
2495		-----		-----	
2590		-----		-----	
2744	EN71-3	320		-0.45	
2751	EN71-3	332.86		-0.20	
2917	EN71-3	359.47		0.31	
2918		-----		-----	
2984		-----		-----	
3013	EN71-3	308		-0.69	
3015	EN71-3	368		0.48	
3116		354.95		0.23	
3124		-----		-----	
3153	EN71-3	359.59		0.32	
3172	EN71-3	426.6		1.62	
3185	EN71-3	362.22		0.37	
3190	EN71-3	341		-0.05	
3195	EN71-3	460		2.27	
3197	EN71-3	355.10		0.23	
3199	EN71-3	185.42	ex	-3.07	test result excluded, see § 4.1
3209	EN71-3	372.41		0.56	
3233	EN71-3	347.26		0.08	
3243	EN71-3	286.138		-1.11	
8005		-----		-----	
					Only test results that maintain pH between 1.1 – 1,3
	normality	OK			suspect
	n	33			27
	outliers	1 + 2ex			1
	mean (n)	343.333			343.574
	st.dev. (n)	44.0215	RSD = 13%		43.1675 RSD = 13%
	R(calc.)	123.260			120.869
	st.dev.(EN71-3:19+A1:21)	51.4999			51.5361
	R(EN71-3:19+A1:21)	144.200			144.301





APPENDIX 2

Determination of migration of other elements on plaster sample #23555; results in mg/kg

lab	As	Ba	B	Cd	Cr (III)
110	not detected	5.762	10.223	not detected	not detected
339	0.520	10.181	8.393	0.267	0.288
551	1.504	7.328	4.59	0.098	0.773
841	0.46	5.8	3.3	0.105	0.327
2118	0.27	4.85	4.60	0.10	0.18
2129	0.420	9.371	10.657	0.127	0.316
2132	<0.25	<25	<25	<0.25	<1
2184	not detected	5.51	not detected	0.25	not detected
2230	0.247	3.80	3.08	-----	-----
2236	<2.0	4.79	6.20	<1.0	<2.0
2250	not detected	4.939	not detected	0.1367	0.2603
2256	1.29	6.85	<2.0	<0.10	0.17
2326	ND	ND	ND	ND	ND
2347	<0.5	<50	<50	<0.5	<5
2350	< 0.5	< 50	< 50	< 0.2	< 5
2366	<0.5	<50	<50	<0.5	<1.0
2372	0.587	<50	<50	0.106	<5
2375	<0.5	<50	<50	0.1	-<5
2381	-----	-----	-----	-----	-----
2385	<1	5.00	4.95	<0.5	<1
2429	<1.0	<10	<50	<0.5	<5
2495	-----	-----	-----	-----	-----
2590	< L.O.Q.	4.42	-----	< L.O.Q.	< L.O.Q.
2744	not detected	not detected	6.5	not detected	not detected
2751	Not Detected	not detected	6.32	Not Detected	Not Detected
2917	0.45	7.10	4.21	0.10	0.21
2918	0.891	-----	-----	0.124	0.41
2984	not detected	not detected	-----	not detected	not analyzed
3013	0.47	6.9	8.1	0.13	0.4092
3015	<1	<10	<50	<0.5	<5
3116	0.390	6.99	6.85	<0.15	<1
3124	-----	-----	-----	-----	-----
3153	< 1	< 10	< 50	< 0.5	< 5
3172	< 1	< 50	< 50	0.158	< 10
3185	<1	<10	<50	<0.5	<5
3190	＜1.0	＜10	＜50	＜0.5	＜5
3195	0.474	6.26	10.8	0.152	not analyzed
3197	<0.5	<10	<10	<0.25	<1
3199	0.205	3.59	3.65	None detected	0.173
3209	<1.0	<10.0	<10.0	<0.1	<5.0
3233	0.46	6.43	9.13	0.60	0.29
3243	0.149	12.521	11.090	0.066	0.159
8005	<2	6.90	-----	<2	-----

Determination of migration of other elements on plaster sample #23555; results in mg/kg
--continued--

lab	Cr (VI)	Co	Hg	Ni
110	not detected	not detected	not detected	not detected
339	not analyzed	0.805	not detected	not detected
551	-----	1.277	0.0036	1.025
841	<0.002	0.88	<0.1	<2.5
2118	0	0.59	0	0.30
2129	<0,02	0.981	<0,1	<1
2132	<0.005	1.15	<0.25	<2.5
2184	not detected	0.70	not detected	not detected
2230	-----	-----	-----	-----
2236	Not Analyzed	<2.0	<2.0	<2.0
2250	<0,02	not detected	not detected	not detected
2256	ND	<1.00	<0.02	0.77
2326	ND	ND	ND	ND
2347	<0.01	1.0	<0.5	<10
2350	< 0.01	0.85	< 0.5	< 10
2366	<0.010	1.0	<0.5	<10
2372	<0.01	1.02	<0.5	<10
2375	<0.01	1.3	<0.5	<10
2381	-----	-----	-----	-----
2385	<0.02	<1	<1	<1
2429	<0.01	<1.0	<1.0	<10
2495	-----	-----	-----	-----
2590	-----	< L.O.Q.	< L.O.Q.	< L.O.Q.
2744	not detected	0.65	not detected	0.8
2751	Not Detected	0.72	Not Detected	0.77
2917	-----	0.49	-----	0.32
2918	-----	1.33	not detected	0.297
2984	not analyzed	-----	not detected	-----
3013	0.7908	1.2	not detected	0.80
3015	<0.01	<1	<1	<10
3116	<0.002	<2	<0.15	<2
3124	-----	-----	-----	-----
3153	< 0.01	< 1	< 1	< 10
3172	< 0.005	< 5	< 5	< 10
3185	<0.01	1.05	<1	<10
3190	＜0.01	＜1.0	<1.0	<10
3195	not analyzed	1.31	<0,10	<2,0
3197	<0.0025	1.01	<0.5	<1
3199	Not tested	None detected	None detected	None detected
3209	<0.01	<1.0	<1.0	<1.0
3233	< 0.0025	0.83	< 0.5	< 5
3243	0	0.710	-0.053	0.243
8005	-----	-----	<2	-----

Determination of migration of other elements on plaster sample #23555; results in mg/kg
--continued--

lab	Se	Sn	Org.Sn	Zn
110	not detected	not detected	not analyzed	15.152
339	not detected	not detected	not analyzed	3.203
551	0.5888	0.0162	-----	5.402
841	<0.5	<0.025	<0.04	<2.5
2118	0.35	0.01	not analyzed	2.14
2129	<1	<0,2	not tested	<10
2132	<2.5	<15	Not detected	<250
2184	not detected	not detected	not detected	not detected
2230	-----	-----	-----	2.27
2236	<2.0	<2.0	Not Analyzed	<50.0
2250	not detected	not detected	not analyzed	not detected
2256	<2	<0.02	ND	<2
2326	ND	ND	ND	ND
2347	<5.0	<0.36	<0.36	<50
2350	< 5	< 0.36	< 0.135	77.9
2366	<5	<0.15	<0.13	<50
2372	<5	<4.9	not analyzed	<50
2375	<5	<0.36	-----	<50
2381	-----	-----	-----	-----
2385	<1	<1	<0.246	<5
2429	<5	<0.1	<0.3	<100
2495	-----	-----	-----	-----
2590	< L.O.Q.	< L.O.Q.	-----	1.99
2744	not detected	not detected	not detected	10
2751	Not Detected	Not Detected	Not Detected	11.98
2917	<0.3	<0.25	-----	<0.25
2918	0.454	not determined	-----	5.54
2984	not detected	-----	-----	-----
3013	0.72	not detected	not detected	6.1
3015	<5	<0.1	<0.3	<100
3116	<2	<2	<0.55	<5
3124	-----	-----	-----	-----
3153	< 5	< 10	< 0.3	< 100
3172	< 10	< 10	-----	< 50
3185	<5	<0.1	<0.3	<100
3190	<5	<0.1	<0.3	<100
3195	0.660	<0,10	not analyzed	<5,0
3197	<1	<10	<0.02	<10
3199	None detected	0.042	Not tested	None detected
3209	<1.0	<0.1	<0.2	<10.0
3233	< 5	< 0.05	-----	< 5
3243	0.078	0.190	-----	2.169
8005	<2	-----	-----	-----

APPENDIX 3
Analytical Details

lab	ISO/IEC17025 accredited	Sample intake (g)	Amount 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 ml	1.6	No	
339	Yes	0.189	9.425	1.41	Yes	1.10
551	Yes	0.25g	12.5mL	1.7	Yes	1.10
841	---	0.2g	10ml	1.25	No	
2118	Yes	0.251 g	12.55ml	2	Yes	1.10
2129	Yes	0,1g	10ml	1,24	Yes	1,14
2132	Yes	0.1000g	5ml	1.14	No	
2184	Yes	0.2g	10ml	1.41	Yes	1.09
2230	Yes	0.1025g	5	1.3	No	
2236	Yes	0.3000	15	1.51	Yes	1.17
2250	Yes	0,1	5	not noted	Yes	not noted
2256	Yes	0.1162g	5.8	1.295	No	
2326	Yes	0.4013 GM	20 ML	1.1 - 1.2	No	
2347	Yes	0.2g	10ml	1.21	No	–
2350	Yes	0.4003 g	20 mL	pH 1.2	No	
2366	Yes				---	
2372	Yes	0.2070g	10mL	1.137	Yes	1.247
2375	Yes	0.1 gram	5 mL	1,15	No	
2381	---				---	
2385	Yes	~0.5 g	Factor 50 to sample intake in mL	1.39	Yes	1.17
2429	Yes	0.1g	5ml	1.16	Yes	1.16
2495	---				---	
2590	Yes	0.1	10	1.52	Yes	1.18
2744	Yes	0,1	5	1,37	Yes	1,10
2751	Yes	0,1021	50	1,46	Yes	1,26
2917	Yes	0.25	12	1.40	Yes	1.10
2918	Yes	0,2057/0,2034/0,0719	10,285/10,17/5,0	1,198/1,152/0,948	No	
2984	Yes	0.2008	10	1.4	No	-
3013	---	0,4597	25		Yes	
3015	Yes	0.1	5	1.2	No	-
3116	Yes	0.5	25		No	
3124	---				---	
3153	Yes	0.1 gram	5 mL	1.44	Yes	1.19
3172	Yes				---	
3185	Yes	0.1030g	5.15mL	1.49	Yes	1.17
3190	Yes	0.1037	5	1.46	Yes	1.16
3195	No	0,10	5	1,1	No	1,1
3197	Yes	0.1479	7.5	1.44	Yes	1.20
3199	Yes	0.1028 g	5.1 mL	1.4	No	na
3209	Yes	0.2001	10	1.50	Yes	1.18
3233	Yes	0.1006	5	1.37	Yes	1.13
3243	Yes	0,5 g	25,5		No	
8005	Yes	0.1	5		---	

APPENDIX 4

Number of participants per country

1 lab in BANGLADESH
1 lab in BELGIUM
1 lab in BRAZIL
1 lab in DENMARK
2 labs in FRANCE
7 labs in GERMANY
5 labs in HONG KONG
1 lab in INDONESIA
3 labs in ITALY
1 lab in KOREA, Republic of
8 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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