Results of Proficiency Test
Total lead in Paint
February 2011

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since the USA Consumer Product Safety Improvement Act (CPSIA) did pass in 2008, iis did receive a number of requests to start a PT scheme for the determination of lead in paint. Among other things, the CPSIA bans lead and phthalates in toys.
This USA legislation reduces the amount of total lead content in the substrates of children's products to 600 ppm by 10 February 2009, to 300 ppm by 14 August 2009 and to 100 ppm by 14 August 2011 and the total lead content in surface coatings or paint to 90 mg/kg by 14 August 2009.

In the 2011 interlaboratory study on total lead in paint 87 laboratories in 24 different countries participated. See appendix 3 for the number of participants per country.
In this report the results of this proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies in Spijkenisse was the organiser of this proficiency test. Sample preparation and analyses were subcontracted.
It was decided to use 2 samples of paint with different concentrations (one high and one low) of lead in this round.
Participants were requested to report results with one extra figure. These unrounded results were preferably used for the statistical evaluations.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO guide 43, ILAC-G13:2007 and ISO 17043:2010. This ensures 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 organisation 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 January 2010 (iis-protocol, version 3.2).

2.3 CONFIDENTIALITY STATEMENT

All data present 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

The materials used in this proficiency test were prepared by a subcontractor by the addition of lead oxide to a regular paint purchased in China. After thorough mixing, the paint was applied to a plastic sheet. After drying, the paint was scraped off the sheet. The dried paint was milled until the particles passed through a 0.5 mm sieve.
The two sieved paint samples, labelled #11008 and #11009 were both divided over 150 subsamples of 0.5 gram each. The samples were tested for homogeneity on 6 randomly selected samples. The analytical testing was performed by a subcontracted laboratory. See the following tables for the homogeneity test results.

	Lead conc. in mg/kg
Sample #11008-1	105
Sample #11008-2	97
Sample #11008-3	105
Sample #11008-4	104
Sample #11008-5	103
Sample #11008-6	103

table 1: measured lead contents for homogeneity test of subsamples #11008

	Lead conc. in mg/kg
Sample #11009-1	567
Sample #11009-2	554
Sample #11009-3	540
Sample #11009-4	554
Sample #11009-5	539
Sample #11009-6	561

table 2: measured lead contents for homogeneity test of subsamples #11009

From the test results of table 1, the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Lead conc. in mg/kg #11008	*Lead conc. in mg/kg #11009*
r (observed)	8.4	31.3
Reference method	Horwitz	Horwitz
0.3 * R (ref. method)	6.9	28.7

table 3: evaluation of the observed repeatabilities of subsamples #11008 and #11009

The calculated repeatabilities are both almost equal to 0.3 times the corresponding reproducibility estimated from the Horwitz equation. Therefore, homogeneity of the subsamples of #11008 and #11009 was assumed.

Approx. 0.5 grams of each of the samples #11008 and #11009 were sent to the participating laboratories on February 16, 2011.

2.5 ANALYSES

The participants were asked to determine the concentration of total lead, applying the analysis procedure that is routinely used in the laboratory and also to treat the PT sample in the way it would normally do with a regular sample in day-to-day circumstances.
To get comparable results a detailed report form, was sent together with the set of samples. On the report forms, the requested total lead content, including the unit and some questions about the analytical details used, were pre-printed. Also a letter of instructions was sent along.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated in the appendices of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that had not yet reported. Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test, see lit.5) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected data are placed under 'Remarks' in the result tables in appendix 1. A list of abbreviations used in the tables can be found in appendix 4.

3.1 STATISTICS

Statistical calculations were performed as described in the report ’iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of January 2010 (iis-protocol, version 3.2)
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as ‘<…’ or ‘>…” were not used in the statistical evaluation.
Before further calculations, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. In the case of an anormal distribution, the statistical evaluation should be used with care.

According to ISO 5725 (1986 and 1994, lit.8 and 9) the original results per determination were submitted subsequently to Dixon’s and Grubbs' outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ 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. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

3.2 GRAPHICS

In order to visualise 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 analysis results are plotted. The corresponding laboratory numbers are under 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 standard. 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 (see appendix 4, nr.13-14).

3.3 Z-SCORES

To evaluate the performance of the individual participating laboratories the z-scores were calculated.
In order to be able to have an objective evaluation of the performance of the individual participants, it was decided to evaluate this performance against the literature requirements. Therefore, the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility by division with 2.8.

The $z_{(target)}$-scores were calculated according to:

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

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

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

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

4 EVALUATION

During the execution of this proficiency test, no problems were encountered.
Only one laboratory decided not to report any results. All other laboratories reported results before the final reporting date.
Finally, the 86 reporting laboratories did report in total 172 numerical results. Observed were 5 statistically outlying results, which is 2.8% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For both samples a Gaussian distribution was found.
Due to the lack of precision data in the relevant test methods for the determination of lead in paint, the z-scores and the calculated reproducibilities were compared with the estimated reproducibility calculated using the Horwitz equation.

4.1 EVALUATION PER SAMPLE

In this section, the determination is discussed. All statistical results reported on the samples are summarised in appendix 1.

Sample #11008: The total lead determination on this sample, at a concentration level of 106 mg/kg, may be somewhat problematic. Only two statistical outliers were observed. However, the observed reproducibility is, after rejection of the statistical outliers, is not in agreement with the target reproducibility estimated from the Horwitz equation. Separate evaluation of the 49 reported SPSC test results leads to the same conclusion.

Sample #11009: The total lead determination on this sample, at a concentration level of 544 mg/kg, was problematic. Three statistical outliers were observed. And the observed reproducibility, after rejection of the statistical outliers, is not in agreement with the target reproducibility estimated from the Horwitz equation. Separate evaluation of the 49 reported SPSC test results leads to the same conclusion.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the target reproducibilities calculated from the Horwitz equation and the reproducibilities as found for the group of participating laboratories. The number of significant results, the average results, the calculated reproducibilities (standard deviation*2.8) and the target reproducibilities are compared in the next table.

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Lead #11008	mg/kg	84	106.0	27.5	23.5
Lead #11009	mg/kg	83	544.4	125.1	94.5

table 4: reproducibilities of lead in paint samples #11008 and #11009

From the above table it can be concluded, without statistical calculations, that the several of the participating laboratories may have some difficulties with the analysis of total lead in

paint when compared with the strict target results calculated with the Horwitz equation. See also the discussions in paragraphs 4.1 and 5.

4.3 COMPARISON WITH PREVIOUS INTERLABORATORY STUDIES

	February 2011	*February 2010*	*February 2009*
Number of reporting labs	86	111	88
Number of results reported	172	222	176
Number of statistical outliers	5	11	10
Percentage outliers	2.8%	4.7%	5.4%

table 5: comparison with previous proficiency tests

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

The evolution of the reproducibility as observed in this proficiency scheme and the comparison with the findings in previous rounds are summarized in table 6.

Range	50-400 mg Pb/kg	400-900 mg Pb/kg
2009	22%	20%
2010	21%	21%
2011	25%	23%
Horwitz' target	18-25%	16-18%

Table 6: comparison of the relative reproducibilities (in %) in the previous PTs and in the present PT

5 DISCUSSION

A large number of different test methods were used. Most often CPSC-CH-E1003-09 was used (49 times) and ASTM E1645 (8 times), followed by 'in house' test methods. Remarkably, most laboratories used the samples 'as received'. Only 11 laboratories did mill (or powder) the samples prior to subsampling for testing. However, as the relative spread for the samples was 23-25%, obviously no significant effect was present on the spread by the differences in pretreatment. This is due to the fact that both sample materials were homogeneous. In real world samples this may be very different.

Most laboratories used microwave digestion using HNO_3 in acc. with CPSC-CH-E1003-09 (and AOAC) or a mixture of HNO_3 with H_2O_2 or HCl in acc. with ASTM E1645 (see appendix 2 for reported details). Only two laboratories used XRF as test method.

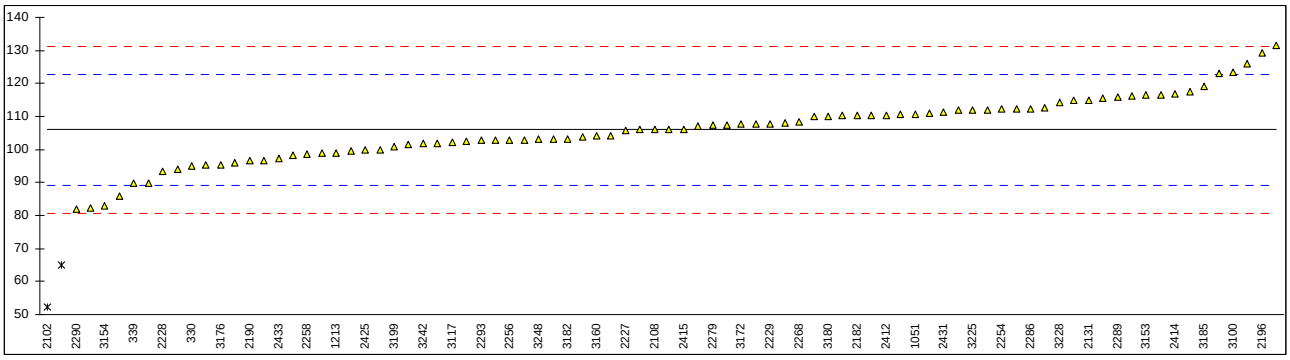
The spreads observed in this interlaboratory study are not caused by just one critical point in the analysis. Consequently, the observed reproducibilities cannot be improved by only one change in the analysis. Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase the quality of the analytical results.

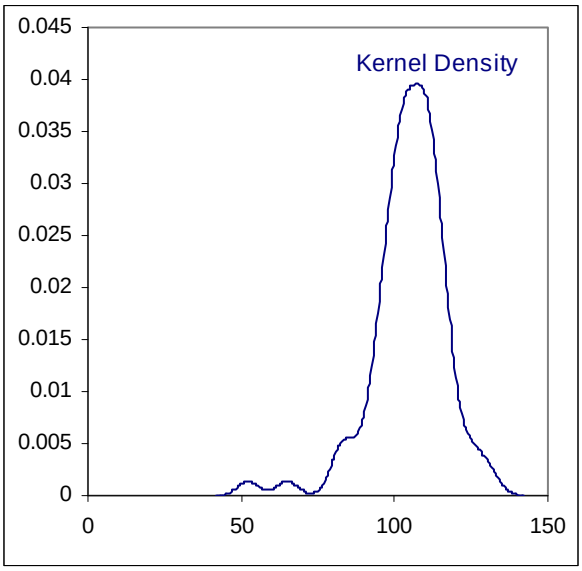
APPENDIX 1

Determination of Total Lead as Pb on sample #11008; results in mg/kg

lab	method	value	mark	z(targ)	remarks
310	in house	103		-0.36	
330	ICP-OES	95		-1.31	
339	ICP	89.7		-1.94	
357	CPSC-CH-E1003-09	106		0.00	
452		-----		-----	
1051	CPSC-CH-E1003-09	110.54		0.54	
1173	XRF	<2000		-----	
1213	ASTM E1645	99.066		-0.82	
2102	in house	52.393	G(0.01)	-6.38	
2108	CPSC	106		0.00	
2118	ICP-OES	125.82		2.36	
2129	ICP-MS	98.82		-0.85	
2131	CPSC-US	115.01	C	1.07	first reported 538.30
2132	CPSC-CH-1003-09	122.91		2.01	
2156	CPSC-CH-1003-09	131.6		3.05	
2160	CPSC-CH-1003-09	100.0		-0.71	
2165	CPSC-CH-1003-09	116.54		1.26	
2172	CPSC-CH-1003-09	110.4		0.53	
2173	CPSC-CH-1003-09	107.3		0.16	
2182	CPSC-CH-1003-09	110.399		0.53	
2190		96.5		-1.13	
2196	CPSC-CH-1003-09	129.1		2.75	
2201	CPSC-CH-1003-09	110.3		0.51	
2214	XRF	103.71		-0.27	
2215	AOAC 974.2	102.5		-0.41	
2225	CPSC-E1003	112.4		0.76	
2227	16 CFR 1303	105.8		-0.02	
2228	16 CFR 1303	93.412		-1.50	
2229	acid digestion	107.8	C	0.22	first reported 134.5
2236	CPSC-CH-1003-09	102.8		-0.38	
2238	ASTM E1645	108		0.24	
2246	in house	109.87		0.46	
2247	CPSC-CH-1003-09	98.3		-0.91	
2253	CPSC-CH-1003-09	112.023		0.72	
2254	microwave digestion	112.25		0.75	
2255	ASTM E1645/E1613	95.2		-1.28	
2256	CPSC-CH-1003-09	102.9		-0.37	
2258	CPSC-CH-1003-09	98.57		-0.88	
2266	16 CFR 1303	110.5		0.54	
2268		108.4		0.29	
2279	CPSC-CH-1003-09	107.3		0.16	
2284	EPA3052	104.1		-0.22	
2286	CPS-CH-E1003-09	112.4		0.76	
2287	ASTM E1645	95.96		-1.19	
2289	CPSC-CH-1003-09	116		1.19	
2290	CPSC-CH-1003-09	81.83		-2.87	
2293	CPSC-CH-1003-09	102.670		-0.39	
2294	CPSC-CH-1003-09	82.3		-2.82	
2295	16 CFR 1303	86	C	-2.38	first reported 45 (EN1122)
2301	CPSC-CH-1003-09	99.47		-0.78	
2407	ASTM E1645	111.8		0.69	
2410	CPSC-CH-1003-09	93.919		-1.44	
2412	CPSC-CH-1003-09	110.4		0.53	
2413	CPSC-CH-1003-09	102.95		-0.36	
2414	ASTM E1645	117		1.31	
2415	CPSC-CH-1003-09	106.07		0.01	
2424	CPSC-CH-1003-09	112.6		0.79	
2425	CPSC-CH-1003-09	100.0		-0.71	
2426	CPSC-CH-1003-09	101.433		-0.54	
2431	ASTM E1645	111.4		0.64	
2433	ASTM E1645	97.14		-1.05	
3100	CPSC-CH-1003-09	123.5		2.08	
3104	EPA 3050B	96.7932		-1.09	
3107	16 CFR 1303	117.6		1.38	
3116	CPSC-CH-1003-09	105.96		0.00	
3117	CPSC-CH-1003-09	102.33	C	-0.43	first reported 142.45
3124	EPA 3052	111		0.60	
3153	CPSC-CH-1003-09	116.4		1.24	
3154	EN1122	82.96		-2.74	
3159	CPSC-CH-1003-09mod	116.3		1.23	
3160	CPSC-CH-1003-09	104.07		-0.23	
3163	XRF	65	C,G(0.01)	-4.88	first reported 303
3166	EPA 200.8	102		-0.47	
3167	CPSC-CH-1003-09	89.81		-1.92	
3169	CPSC-CH-1003-09	115.63		1.15	

3172	CPSC-CH-1003-09	107.6		0.19	
3176	16 CFR 1303	95.4	C	-1.26	first reported 150.9
3180	microwave digestion	110	C	0.48	first reported 69
3182	CPSC-CH-1003-09	103.09		-0.34	
3185	CPSC-CH-1003-09	119.2		1.57	
3190	CPSC-CH-1003-09	107		0.12	
3199	in house	100.9		-0.60	
3210	CPSC-CH-1003-09	115.0		1.07	
3218	CPSC-CH-1003-09	107.7		0.20	
3225	CPSC-CH-1003-09	111.9		0.70	
3228	acid digestion	114.4		1.00	
3242	CPSC-CH-1003-09	102		-0.47	
3248	CPSC	103		-0.36	
					only CPSC data:
	normality	OK			OK
	n	84			49
	outliers	2			0
	mean (n)	105.98			107.56
	st.dev. (n)	9.802			9.868
	R(calc.)	27.45			27.63
	R(Horwitz)	23.53			23.83

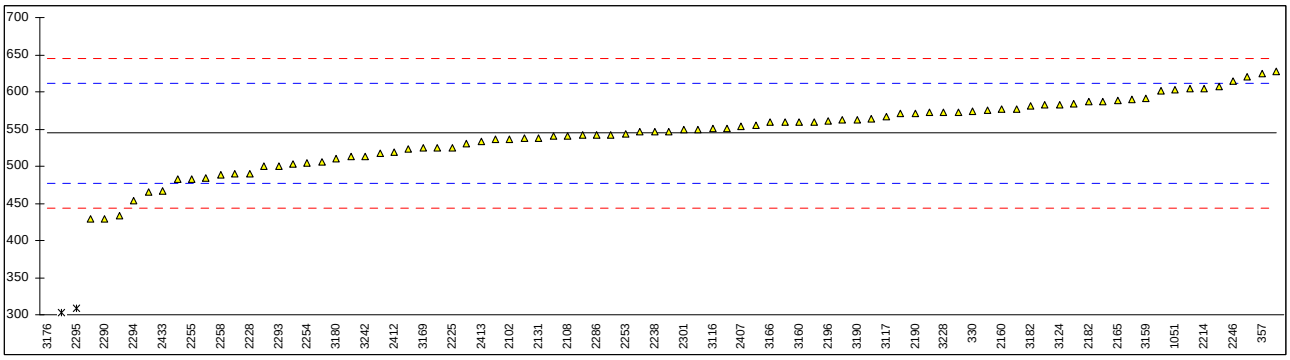


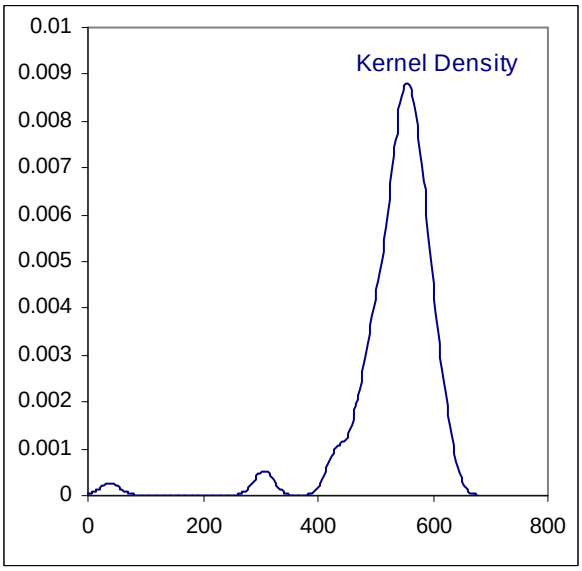


Determination of Total Lead as Pb on sample #11009; results in mg/kg

lab	method	value	mark	z(targ)	remarks
310	in house	559		0.43	
330	ICP-OES	574		0.88	
339	ICP	562		0.52	
357	CPSC-CH-E1003-09	625		2.39	
452		-----		-----	
1051	CPSC-CH-E1003-09	602.50		1.72	
1173	XRF	<2000		-----	
1213	ASTM E1645	502.366		-1.24	
2102	in house	536.926		-0.22	
2108	CPSC	541		-0.10	
2118	ICP-OES	551.12		0.20	
2129	ICP-MS	518.00		-0.78	
2131	CPSC-US	538.30	C	-0.18	first reported 115.01
2132	CPSC-CH-1003-09	589.39		1.33	
2156	CPSC-CH-1003-09	464.7		-2.36	
2160	CPSC-CH-1003-09	576.4		0.95	
2165	CPSC-CH-1003-09	588.15		1.30	
2172	CPSC-CH-1003-09	546.2		0.05	
2173	CPSC-CH-1003-09	620.1		2.24	
2182	CPSC-CH-1003-09	586.979		1.26	
2190		571.1		0.79	
2196	CPSC-CH-1003-09	561.2		0.50	
2201	CPSC-CH-1003-09	547.1		0.08	
2214	XRF	605.04		1.80	
2215	AOAC 974.2	540.0		-0.13	
2225	CPSC-E1003	525.1		-0.57	
2227	16 CFR 1303	541.6		-0.08	
2228	16 CFR 1303	489.606		-1.62	
2229	acid digestion	627.3		2.46	
2236	CPSC-CH-1003-09	587.6		1.28	
2238	ASTM E1645	547		0.08	
2246	in house	614.21		2.07	
2247	CPSC-CH-1003-09	531.1		-0.39	
2253	CPSC-CH-1003-09	542.998		-0.04	
2254	microwave digestion	504.80		-1.17	
2255	ASTM E1645/E1613	482.5		-1.83	
2256	CPSC-CH-1003-09	563.4		0.56	
2258	CPSC-CH-1003-09	488.43		-1.66	
2266	16 CFR 1303	428.5		-3.43	
2268		524.8		-0.58	
2279	CPSC-CH-1003-09	542.5		-0.05	
2284	EPA3052	536.2		-0.24	
2286	CPS-CH-E1003-09	541.8		-0.08	
2287	ASTM E1645	601.03		1.68	
2289	CPSC-CH-1003-09	537		-0.22	
2290	CPSC-CH-1003-09	429.55		-3.40	
2293	CPSC-CH-1003-09	500.700		-1.29	
2294	CPSC-CH-1003-09	453.6		-2.69	
2295	16 CFR 1303	309	C,G(0.01)	-6.97	first reported 140 (EN1122)
2301	CPSC-CH-1003-09	549.10		0.14	
2407	ASTM E1645	553.3		0.27	
2410	CPSC-CH-1003-09	512.65		-0.94	
2412	CPSC-CH-1003-09	519.3		-0.74	
2413	CPSC-CH-1003-09	533.03		-0.34	
2414	ASTM E1645	577		0.97	
2415	CPSC-CH-1003-09	522.67		-0.64	
2424	CPSC-CH-1003-09	505.76		-1.14	
2425	CPSC-CH-1003-09	489.25		-1.63	
2426	CPSC-CH-1003-09	482.268		-1.84	
2431	ASTM E1645	483.9		-1.79	
2433	ASTM E1645	467.25		-2.28	
3100	CPSC-CH-1003-09	573.0		0.85	
3104	EPA 3050B	574.955		0.91	
3107	16 CFR 1303	582.25		1.12	
3116	CPSC-CH-1003-09	550.07		0.17	
3117	CPSC-CH-1003-09	566.32		0.65	
3124	EPA 3052	583		1.15	
3153	CPSC-CH-1003-09	571.8		0.81	
3154	EN1122	433.0		-3.30	
3159	CPSC-CH-1003-09mod	591.4		1.39	
3160	CPSC-CH-1003-09	559.20		0.44	
3163	XRF	303	C,G(0.01)	-7.15	first reported 65
3166	EPA 200.8	559		0.43	
3167	CPSC-CH-1003-09	570.7		0.78	
3169	CPSC-CH-1003-09	524.47		-0.59	
3172	CPSC-CH-1003-09	607.4		1.87	
3176	16 CFR 1303	38.1	C,G(0.01)	-15.00	first reported 756.8

3180	microwave digestion	510	-1.02	
3182	CPSC-CH-1003-09	580.90	1.08	
3185	CPSC-CH-1003-09	584.1	1.18	
3190	CPSC-CH-1003-09	562	0.52	
3199	in house	554.4	0.30	
3210	CPSC-CH-1003-09	500.0	-1.31	
3218	CPSC-CH-1003-09	560.1	0.47	
3225	CPSC-CH-1003-09	604	1.77	
3228	acid digestion	572.6	0.84	
3242	CPSC-CH-1003-09	513	-0.93	
3248	CPSC	550	0.17	
				only CPSC data:
	normality	OK		OK
	n	83		49
	outliers	3		0
	mean (n)	544.35		546.70
	st.dev. (n)	44.662		42.815
	R(calc.)	125.05		119.88
	R(Horwitz)	94.49		94.83





APPENDIX 2

Analytical details as used by the participants

Lab	Digestion technique	Pretreatment	Remarks
310	microwave	as received	Nitric Acid
330	microwave	as received	ICP/OES
339			
357	microwave	as received	
452			
1051	microwave	as received	
1173	XRF		detection limit of method is 0.2%
1213	microwave	as received	
2102	acid digestion	as received	
2108	microwave	as received	
2118	acid digestion	as received	HNO3/H2O2 and there was some presence of TiO2
2129	microwave		MWS-Ultraclave
2131	microwave		
2132	microwave	as received	
2156	acid digestion	powdered	ICP/OES
2160	microwave	as received	Detection with EDXRF/Quantification with AAS
2165	microwave	as received	
2172	acid digestion	as received	
2173	acid digestion	as received	
2182	microwave	as received	
2190	hot plate		
2196	acid digestion	powdered	
2201	hot plate	as received	
2214			
2215	hot plate	milled	
2225	microwave	as received	
2227	hot plate	2 hrs	drying in oven @105°C
2228	hot plate	as received	
2229	acid digestion	powered	
2236	microwave	as received	
2238	microwave	as received	
2246	microwave	as received	HNO3/HF/H2O2
2247	microwave	as received	
2253	microwave	as received	
2254	microwave	as received	Nitric Acid
2255	acid digestion	as received	
2256	microwave	as received	
2258	hot plate	as received	
2266	microwave	as received	needed more sample as QC failed
2268	microwave	as received	
2279	acid digestion	as received	
2284	microwave	powdered	
2286	microwave	as received	
2287	microwave	powdered	
2289	acid digestion	as received	
2290	microwave	as received	
2293	acid digestion	as received	
2294	microwave	as received	
2295	microwave	as received	
2301	microwave	as received	
2407	microwave	as received	
2410	microwave	as received	
2412	microwave	as received	
2413	microwave	as received	
2414	microwave	as received	

2415	microwave	as received	Nitric Acid
2424	microwave	as received	
2425	microwave	as received	
2426	microwave	as received	
2431	hot plate	as received	
2433	acid digestion	as received	
3100	microwave	as received	
3104	acid digestion	as received	
3107	microwave	as received	Nitric Acid
3116	microwave	as received	
3117	microwave	powdered	
3124	microwave	milled	6ml HN03, 2ml HCl, 1ml HF to 0.25g sample
3153	microwave	as received	ICP/OES
3154	microwave	as received	
3159	microwave	as received	
3160	microwave	as received	
3163	XRF	as received	
3166	microwave	as received	
3167	microwave	as received	
3169	microwave	as received	
3172	microwave	as received	
3176	microwave	powdered	mix of HNO3, H2SO4, H2O2, HF
3180	microwave		
3182	microwave	as received	
3185	microwave	as received	
3190	microwave	as received	
3199	hot plate		Nitric Acid
3210	microwave		
3218	microwave	as received	
3225	microwave	as received	
3228	acid digestion	sieved	
3242	microwave	as received	
3248	microwave	powdered	

APPENDIX 3

Number of participants per country

2 labs in	BANGLADESH
1 lab in	BELGIUM
1 lab in	DENMARK
1 lab in	FINLAND
5 labs in	FRANCE
3 labs in	GERMANY
2 labs in	GUATEMALA
11 labs in	HONG KONG
2 labs in	INDIA
1 lab in	ISRAEL
1 lab in	ITALY
2 labs in	JAPAN
1 lab in	KOREA
1 lab in	MALAYSIA
2 labs in	MEXICO
26 labs in	P.R. of CHINA
1 lab in	PAKISTAN
1 lab in	PHILIPPINES
1 lab in	SPAIN
2 labs in	SWITZERLAND
1 lab in	THAILAND
3 labs in	THE NETHERLANDS
3 labs in	TURKEY
8 labs in	U.S.A.
3 labs in	UNITED KINGDOM
3 labs in	VIETNAM

APPENDIX 4

Abbreviations:

C	= final result after checking of first reported suspect 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
n.a.	= not applicable
n.d.	= not detected
n.r.	= not reported

Literature:

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