Results of Proficiency Test
Total lead in Paint
February 2012

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. L.Dijkstra
Correctors: ing. R.J. Starink & dr. R.G. Visser

Report: iis12V01

April 2012

CONTENTS

Appendices:

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 2012 interlaboratory study on total lead in paint 112 laboratories in 33 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 low and one high) of lead in this round.
Participants were requested to report rounded and unrounded results. 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/IEC 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

Two different paint samples were used in this proficiency test. Each paint sample was prepared by a different company. Two regular paint types were used. However, to the paint for sample #12010 lead nitrate was added and to the paint for sample #12011 lead oxide was added.
After thorough mixing, both paint samples were applied to plastic sheets. After drying, the paint was scraped off the sheets. The dried paint was milled until the particles passed through a 0.5 mm sieve.
The two dried and sieved paint samples, labelled #12010 and #12011 were respectively divided over 129 and 140 subsamples of 0.5 gram each. The samples, labelled #12010 were tested for homogeneity on 5 randomly selected samples and the samples labelled #12011, were tested for homogeneity on 4 randomly selected samples and the 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 #12010-1	38.1
Sample #12010-2	37.7
Sample #12010-3	37.4
Sample #12010-4	37.6
Sample #12010-5	38.0

table 1: homogeneity test results of subsamples #12010

	Lead conc. in mg/kg
Sample #12011-1	303
Sample #12011-2	295
Sample #12011-3	315
Sample #12011-4	283

table 2: : homogeneity test results of subsamples #12011

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 #12010	Lead conc. in mg/kg #12011
r (observed)	0.81	37.7
Reference method	Horwitz	Horwitz
0.3 * R (ref. method)	2.9	17.0

table 3: repeatabilities of subsamples #12010 and subsamples #12011

The calculated repeatabilities for samples #12010 and #12011 are respectively in good and in not good agreement with 0.3 times the estimated target reproducibilities, calculated using the Horwitz equation. However, the calculated repeatability for sample #12011 is in agreement with the usual repeatability of the laboratory that performed the homogeneity tests.
Therefore, homogeneity of subsamples #12010 and #12011 was assumed.

Approx. 0.5 grams of each of the samples #12010 and #12011 were sent to the participating laboratories on February 16, 2012.

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.

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. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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 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, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

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 the fit-for-useness of the reported test result.

In case no literature reproducibility was available, other target values were used. In some cases literature repeatability is available; in other cases a reproducibility of a former iis proficiency test could be used and also the Horwitz equation can be used to estimate target reproducibility.

The z-scores were calculated according to:

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

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

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

4 EVALUATION

In this proficiency test, some problems were encountered with despatch of the samples. Of the 112 participants, 21 participants reported results after the final reporting date and two laboratories reported no results at all.
Finally, the 110 reporting laboratories did report in total 215 numerical results. Observed were 9 statistically outlying results, which is 4.2% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For both samples a not normal Gaussian distribution was found. Therefore the statistical evaluation for both sampled should be used with due care.
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 #12010: The total lead determination on this sample, at a low concentration level of 39 mg/kg, may be problematic for a number of laboratories. Six statistical outliers were observed. The observed reproducibility is, after rejection of the statistical outliers, in full agreement with the target reproducibility estimated from the Horwitz equation. When the 65 reported SPSC test results are evaluated seperately then the observed reproducibility is also in agreement with the target reproducibility estimated from the Horwitz equation.

Sample #12011: The total lead determination on this sample, at a medium concentration level of 274 mg/kg, may be 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. When the 65 reported SPSC test results are evaluated separately, the observed reproducibility is smaller but still not in agreement with the target reproducibility estimated from the Horwitz equation.

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 #12010	mg/kg	100	38.7	10.6	10.0
Lead #12011	mg/kg	106	274.4	76.5	52.8

table 4: reproducibilities of lead in paint samples #12010 and #12011

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 EVALUTION OF THE PROFICIENCY TEST OF FEBRUARY 2012 WITH PREVIOUS PTS

	February 2012	*February 2011*	*February 2010*	*February 2009*
Number of reporting labs	110	86	111	88
Number of results reported	215	172	222	176
Number of statistical outliers	9	5	11	10
Percentage outliers	4.2%	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	30-300 mg Pb/kg	300-900 mg Pb/kg
2009	22%	20%
2010	n.e.	21 - 22%
2011	26%	23%
2012	27 - 28%	n.e.
Horwitz' target	19 - 27%	16 - 19%

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 (65 times). ASTM E1645 was used 6 times and the EPA 3050B /EPA 3052B were both used 1 time. Other laboratories used 'in house' test methods.
Remarkably, most laboratories used the samples 'as received'. Only 12 laboratories did mill (or sieve) the samples prior to subsampling for testing. However, as the relative spread for the samples was 27-28%, 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 in acc. with CPSC-CH-E1003-09 (and AOAC). Other laboratories mentioned as digestion technique "acid digestion". Only two laboratories used XRF as test method.

The differences in quality of the test results of the two samples may be explained by the differences in lead concentration, but will also be caused by the differences in the type of paint used and the type of lead compound used. But obviously the effects of these differences are all rather small for the samples used in this proficiency test.

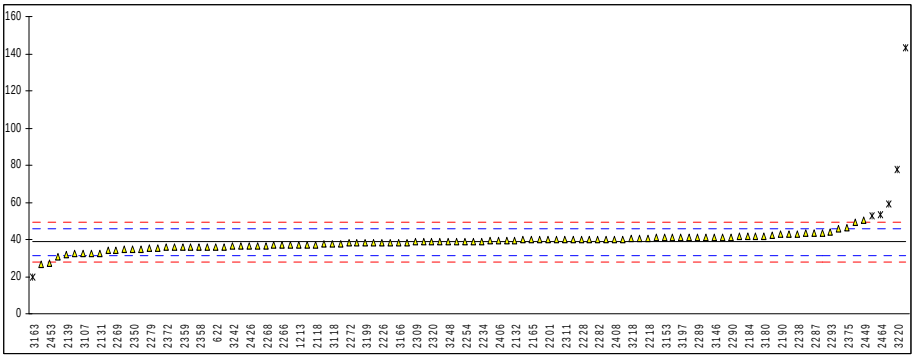
The spreads observed in this interlaboratory study are not caused by just one critical point in the analysis. Consequently, the observed reproducibilities cannot be improved by only one change in the analysis. Each laboratory has to evaluate its performance in this study and make decisions about necessary 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.

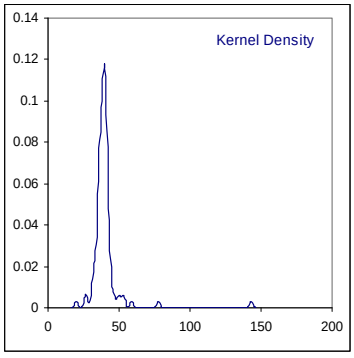
APPENDIX 1

Determination of Total Lead as Pb on sample #12010; results in mg/kg

lab	Method	value	mark	z(targ)	remarks
310	in house	36.9		-0.50	
330		31		-2.15	
357	CPSC-CH-E1003-09	46		2.05	
551	CPSC-CH-E1003-09	26.71		-3.35	
622	in house	36.1	C	-0.72	first reported: 72.1
632		-----		-----	
1213	CPSC-CH-E1003-09	37.18		-0.42	
2102		58.95	G(0.01)	5.68	
2118	in house	37.19		-0.42	
2129	in house	43.23		1.28	
2131	CPSC-US	32.60		-1.70	
2132	CPSC-CH-E1003-09	39.53		0.24	
2139	ASTM E1645	32		-1.87	
2156	CPSC-CH-E1003-09	32.4		-1.76	
2165	CPSC-CH-E1003-09	39.8		0.31	
2172	CPSC-CH-E1003-09	42.82		1.16	
2184	CPSC-CH-E1003-09	41.5		0.79	
2190	in house	42.7		1.13	
2196	CPSC-CH-E1003-09	40.9		0.62	
2201	ASTM E1645	40.0		0.37	
2217	ASTM E1645/E1613	143.1	G(0.01)	29.25	
2218	in house	40.859		0.61	
2225	CPSC-CH-E1003-09	37.9		-0.22	
2226	CPSC-CH-E1003-09	38.3		-0.11	
2228	CPSC-16CFR-1303	40.108		0.40	
2229	CPSC-CH-E1003-09	34.95		-1.04	
2232	CPSC-CH-E1003-09	40.88		0.62	
2234	CPSC-CH-E1003-09	39.125		0.13	
2236	CPSC-CH-E1003-09	36.3		-0.67	
2238	CPSC-CH-E1003-09	43.0		1.21	
2240	AOAC 974.2	37.58		-0.31	
2245	CPSC-CH-E1003-09	40.15		0.41	
2246	CPSC-CH-E1003-09	34.25		-1.24	
2247	in house	43.6		1.38	
2253	CPSC-CH-E1003-09	39.803		0.32	
2254	in house	39.01		0.09	
2255	CPSC-CH-E1003-09	39.72		0.29	
2256	CPSC-CH-E1003-09	39.02		0.10	
2258	CPSC-16CFR-1303	34.592		-1.14	
2266	CPSC-16CFR-1303	37.0		-0.47	
2268	CPSC-CH-E1003-09	36.8		-0.53	
2269	in house	34.25		-1.24	
2272	CPSC-CH-E1003-09	38.0		-0.19	
2277	in house	n.d.		-----	
2279	CPSC-CH-E1003-09	35.1		-1.00	
2282	in house	40.2		0.43	
2284	CPSC-CH-E1003-09	38.8		0.03	
2286	CPSC-CH-E1003-09	36.7		-0.55	
2287	ASTM E1645	43.5		1.35	
2289	CPSC-CH-E1003-09	41.08		0.67	
2290	CPSC-CH-E1003-09	41.4		0.76	
2293	CPSC-CH-E1003-09	43.870		1.45	
2294	CPSC-CH-E1003-09	52.6	G(0.05)	3.90	
2295	CPSC-CH-E1003-09	40		0.37	
2303	in house	49.34		2.99	
2309	CPSC-CH-E1003-09	38.65		-0.01	
2311	CPSC-CH-E1003-09	40.04		0.38	
2320	CPSC-CH-E1003-09	38.8		0.03	
2350	CPSC-CH-E1003-09	34.84		-1.07	
2358	CPSC-CH-E1003-09	36		-0.75	
2359	CPSC-CH-E1003-09	35.917		-0.77	
2372	in house	35.75		-0.82	
2375	ASTM E1645	46.5		2.19	
2380	CPSC-CH-E1003-09	39.24		0.16	
2390	ASTM E1645	35.5		-0.89	
2406	CPSC-CH-E1003-09	39.44		0.21	
2408	CPSC-CH-E1003-09	40.20805		0.43	
2410	CPSC-CH-E1003-09	39.0		0.09	
2412	CPSC-CH-E1003-09	38.22		-0.13	
2413	CPSC-CH-E1003-09	36.2		-0.69	
2424	CPSC-CH-E1001-8.1	40.456		0.50	
2425	CPSC-CH-E1003-09	35.95		-0.76	
2426	CPSC-CH-E1003-09	36.67		-0.56	

2431	in house	38.4		-0.08	
2433	ASTM E1645	37.18		-0.42	
2449	CPSC-CH-E1003-09	50.5		3.31	
2450	CPSC-CH-E1003-09	40.05		0.38	
2453	CPSC-CH-E1002-08	27		-3.27	
2460		-----		-----	
2463	CPSC-CH-E1003-09	40.255		0.44	
2464	CPSC-CH-E1003-09	53.55	CG(0.05)	4.17	first reported: 99.01
2465	in house	32.58		-1.71	
2471	CPSC-CH-E1003-09	38.8393		0.05	
2480		-----	W	-----	result withdrawn
2482	CPSC-CH-E1003-09	38.04		-0.18	
3100	CPSC-CH-E1003-09	38.33		-0.10	
3107	CPSC-16CFR-1303	32.5		-1.73	
3116	CPSC-CH-E1003-09	41.40		0.76	
3118	CPSC-CH-E1003-09	37.64		-0.29	
3124	EPA3052Mod.	41.3		0.73	
3146	in house	41.4		0.76	
3153	CPSC-CH-E1003-09	40.9		0.62	
3154	EN1122	36.0	C	-0.75	first reported: 91.20
3160	CPSC-CH-E1003-09	41.55		0.80	
3163	in house	20	G(0.01)	-5.23	
3166	in house	38.4		-0.08	
3167	CPSC-CH-E1003-09	41.48		0.79	
3172	CPSC-CH-E1003-09	35.8		-0.81	
3176	CPSC-16CFR-1303	41		0.65	
3180		41.9	C	0.90	first reported: 20.75
3182	CPSC-CH-E1003-09	37.08		-0.45	
3184	CPSC-CH-E1003-09	39.48		0.22	
3190	CPSC-CH-E1003-09	40.2		0.43	
3197	CPSC-CH-E1003-09	40.98		0.65	
3199	AOAC 974.2	38.21		-0.13	
3210	CPSC-CH-E1003-09	<90		-----	
3218	CPSC-CH-E1003-09	40.32		0.46	
3220	EPA3050B	77.7	C,G(0.01)	10.93	first reported: 57.7
3228	CPSC-CH-E1003-09	42.6		1.10	
3242	in house	36.24		-0.68	
3248	CPSC-16CFR-1303	39		0.09	
					SPSC data only:
	normality	not OK			not OK
	n	100			62
	outliers	6			2
	mean (n)	38.677			38.977
	st.dev. (n)	3.7711			3.2888
	R(calc.)	10.559			9.209
	R(Horwitz)	9.995			10.061



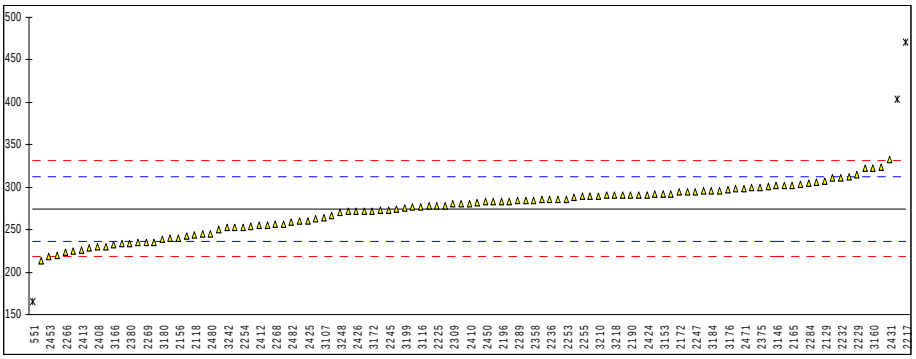


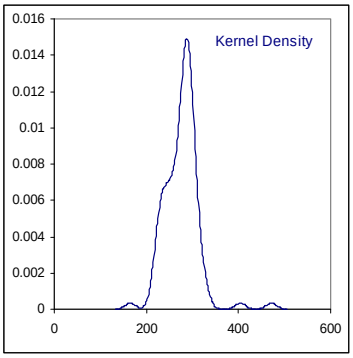
Determination of Total Lead as Pb on sample #12011; results in mg/kg

lab	method	value	mark	z(targ)	remarks
310	in house	281.4		0.37	
330		220		-2.89	
357	CPSC-CH-E1003-09	213		-3.26	
551	CPSC-CH-E1003-09	164.87	G(0.05)	-5.81	
622	in house	228.7	C	-2.43	first reported: 457.2
632		-----		-----	
1213	CPSC-CH-E1003-09	277.8		0.18	
2102		259.78		-0.78	
2118	in house	243.82		-1.62	
2129	in house	307.37		1.75	
2131	CPSC-US	253.00		-1.14	
2132	CPSC-CH-E1003-09	290.47		0.85	
2139	ASTM E1645	272		-0.13	
2156	CPSC-CH-E1003-09	240.4		-1.81	
2165	CPSC-CH-E1003-09	302.4		1.48	
2172	CPSC-CH-E1003-09	294.73		1.08	
2184	CPSC-CH-E1003-09	288.3		0.73	
2190	in house	290.5		0.85	
2196	CPSC-CH-E1003-09	283.6		0.49	
2201	ASTM E1645	299.2		1.31	
2217	ASTM E1645/E1613	471.2	G(0.01)	10.43	
2218	in house	283.487		0.48	
2225	CPSC-CH-E1003-09	278.2		0.20	
2226	CPSC-CH-E1003-09	278.7		0.23	
2228	CPSC-16CFR-1303	305.674		1.66	
2229	CPSC-CH-E1003-09	315.25		2.16	
2232	CPSC-CH-E1003-09	311.1		1.94	
2234	CPSC-CH-E1003-09	271.25		-0.17	
2236	CPSC-CH-E1003-09	285.8		0.60	
2238	CPSC-CH-E1003-09	302		1.46	
2240	AOAC 974.2	235.45		-2.07	
2245	CPSC-CH-E1003-09	273.38		-0.06	
2246	CPSC-CH-E1003-09	266.21		-0.44	
2247	in house	295.1		1.10	
2253	CPSC-CH-E1003-09	286.288		0.63	
2254	in house	253.29	C	-1.12	first reported: 399.95
2255	CPSC-CH-E1003-09	289.45		0.80	
2256	CPSC-CH-E1003-09	294.95		1.09	
2258	CPSC-16CFR-1303	242.18		-1.71	
2266	CPSC-16CFR-1303	224.0		-2.67	
2268	CPSC-CH-E1003-09	256.1		-0.97	
2269	in house	235.26		-2.08	
2272	CPSC-CH-E1003-09	249.7		-1.31	
2277	in house	403.8294	G(0.01)	6.86	
2279	CPSC-CH-E1003-09	253.5		-1.11	
2282	in house	284.5		0.53	
2284	CPSC-CH-E1003-09	304.5		1.59	
2286	CPSC-CH-E1003-09	256.9		-0.93	
2287	ASTM E1645	324.3		2.64	
2289	CPSC-CH-E1003-09	284.2		0.52	
2290	CPSC-CH-E1003-09	290.2		0.84	
2293	CPSC-CH-E1003-09	274.450		0.00	
2294	CPSC-CH-E1003-09	276.2		0.09	
2295	CPSC-CH-E1003-09	240		-1.83	
2303	in house	291.59		0.91	
2309	CPSC-CH-E1003-09	280.47		0.32	
2311	CPSC-CH-E1003-09	298.043		1.25	
2320	CPSC-CH-E1003-09	310.8		1.93	
2350	CPSC-CH-E1003-09	272.8		-0.09	
2358	CPSC-CH-E1003-09	285		0.56	
2359	CPSC-CH-E1003-09	289.645		0.81	
2372	in house	291.3		0.89	
2375	ASTM E1645	299.5		1.33	
2380	CPSC-CH-E1003-09	233.40		-2.18	
2390	ASTM E1645	230.4	C	-2.34	first reported: 195.8
2406	CPSC-CH-E1003-09	286.0		0.61	
2408	CPSC-CH-E1003-09	230.1148		-2.35	
2410	CPSC-CH-E1003-09	281.0		0.35	
2412	CPSC-CH-E1003-09	255.32		-1.01	
2413	CPSC-CH-E1003-09	226.4		-2.55	
2424	CPSC-CH-E1001-8.1	291.3825		0.90	
2425	CPSC-CH-E1003-09	260.04		-0.76	
2426	CPSC-CH-E1003-09	271.62		-0.15	
2431	in house	333		3.10	
2433	ASTM E1645	224.55		-2.65	

2449	CPSC-CH-E1003-09	234.61	-2.11
2450	CPSC-CH-E1003-09	283.01	0.45
2453	CPSC-CH-E1002-08	219	-2.94
2460		-----	-----
2463	CPSC-CH-E1003-09	280.800	0.34
2464	CPSC-CH-E1003-09	303.99	1.57
2465	in house	233.2	-2.19
2471	CPSC-CH-E1003-09	298.2463	1.26
2480	in house	245.6	-1.53
2482	CPSC-CH-E1003-09	259.51	-0.79
3100	CPSC-CH-E1003-09	296.30	1.16
3107	CPSC-16CFR-1303	264.0	-0.55
3116	CPSC-CH-E1003-09	276.91	0.13
3118	CPSC-CH-E1003-09	292.08	0.94
3124	EPA3052Mod.	322	2.52
3146	in house	302	1.46
3153	CPSC-CH-E1003-09	292.0	0.93
3154	EN1122	283.7	0.49
3160	CPSC-CH-E1003-09	322.09	2.53
3163	in house	245	-1.56
3166	in house	232	-2.25
3167	CPSC-CH-E1003-09	255.6	-1.00
3172	CPSC-CH-E1003-09	272.2	-0.12
3176	CPSC-16CFR-1303	297	1.20
3180		238.43	-1.91
3182	CPSC-CH-E1003-09	262.30	-0.64
3184	CPSC-CH-E1003-09	296.3	1.16
3190	CPSC-CH-E1003-09	301	1.41
3197	CPSC-CH-E1003-09	311.80	1.98
3199	AOAC 974.2	276.10	0.09
3210	CPSC-CH-E1003-09	290	0.82
3218	CPSC-CH-E1003-09	290.4	0.85
3220	EPA3050B	285.6	0.59
3228	CPSC-CH-E1003-09	295.3	1.11
3242	in house	252.69	-1.15
3248	CPSC-16CFR-1303	270	-0.24

		SPSC data only:
normality	not OK	not OK
n	106	64
outliers	3	1
mean (n)	274.445	278.502
st.dev. (n)	27.3309	23.3241
R(calc.)	76.526	65.307
R(Horwitz)	52.809	53.472





APPENDIX 2

Analytical details as used by the participants

Lab	Digestion technique	Pretreatment	Remarks
310	microwave	as received	HNO_3; results rounded on EPA 2 measurements by ICPMS
330	microwave	no pretreatment	by microwave oven 0.1g in 25 mL; ICP/OES
357	microwave	as received	
551	microwave	powdered	
622	acid digestion	as received (powder)	
632			
1213	microwave	powdered	
2102	XRF	as received	
2118	acid digestion	as received	HNO_3/H_2O_2;; After resolving the sample (#12010) there was a little white precipate present. Control in presence of lead in white precipitate with XRF nagative. There was some presence of titanium dioxide.
2129	mws ultra clave		
2131	microwave	as received	
2132	microwave	as received	conc. HNO_3
2139	microwave	as received	
2156	acid digestion	as received	
2165	microwave	as received	
2172	acid digestion	as received	
2184	microwave	as received	
2190	acid digestion	as received	dosage by ICP/OES
2196	acid digestion	powdered	
2201	microwave	as received	
2217	microwave	as received	
2218	microwave		acid digestion
2225	microwave	as received	
2226	microwave	as received	
2228	hot plate digestion	as received	
2229	acid digestion	powdered	
2232	acid digestion	as received	
2234	microwave	as received	
2236	microwave	as received	
2238	acid digestion	as received	
2240	acid digestion	as received	
2245	acid digestion		
2246	microwave	as received	
2247	microwave	powdered	
2253	microwave	as received	
2254	microwave	as received	acid digestion
2255	acid digestion	as received	
2256	microwave	as received	
2258	hot plate digestion	as received	HNO3 40%
2266	acid digestion		by microwave & ICP
2268	microwave	as received	
2269	hot block method	as received	
2272	microwave	powdered	not totally dissolve
2277	acid digestion	as received	
2279	microwave	as received	acid digestion
2282	acid digestion	powdered	
2284	microwave	as received	
2286	microwave	as received	
2287	microwave	as received	
2289	microwave	as received	
2290	microwave	as received	HNO3; quantification with ICP/OES
2293	microwave	as received	acid digestion
2294	microwave	as received	
2295	microwave	powdered	

2303	microwave	as received	assisted acid digestion; #12010 difficult to digest
2309	microwave	as received	
2311	microwave	as received	
2320	microwave	as received	
2350	microwave	powdered	
2358	microwave	as received	
2359	acid digestion	as received	
2372	microwave	powdered	
2375	microwave	pretreated	acid digestion
2380	acid digestion	as received	
2390	microwave	as received	acid digestion
2406	microwave	as received	acid digestion
2408	microwave	as received	
2410	microwave		
2412	microwave	sieved	
2413	microwave	as received	acid digestion
2424	microwave	as received	
2425	microwave	as received	HNO_3 & H_2O_2
2426	acid digestion	as received	
2431	hot plate	as received	HNO_3/H_2O_2, Peterence Method, ASTME1645-01
2433	acid digestion	cut into peces	
2449	acid digestion	as received	
2450	acid digestion	powdered	
2453	microwave	as received	#12010 not completely digested
2460			
2463	microwave	as received	acid digestion: ICP/OES
2464	acid digestion	as received	microwave was attempted, but the sample was to fluffy and static to walls.Used acid digestion instead.
2465	microwave	as received	
2471	acid digestion	powdered	
2480	microwave		
2482	microwave	as received	
3100	microwave	as received	
3107	microwave	as received	
3116	microwave		acid digestion
3118			
3124	microwave	as received	acid digestion
3146	microwave	as received	acid digestion
3153	microwave	as received	acid digestion; ICP/OES
3154	microwave	as received	
3160	microwave	as received	
3163	XRF	as received	
3166	microwave	as received	
3167	microwave	as received	
3172	microwave	as received	
3176	microwave	as received	More details about microwave technique (temp, pressure,time) would be more useful to see differences between microwaves.
3180	microwave	as received	
3182	microwave	as received	
3184	microwave	as received	
3190	microwave	as received	
3197	microwave	as received	
3199	hot plate	as received	acid digestion
3210	microwave	as received	HNO_3
3218	microwave	as received	
3220	microwave	as received	
3228	microwave	as received	
3242	hot plate	as received	HNO_3 / H_2O_2
3248	microwave	as received	

APPENDIX 3

Number of participants per country

3 labs in BANGLADESH
1 lab in BELGIUM
1 lab in BRAZIL
2 labs in CANADA
1 lab in DENMARK
1 lab in FINLAND
5 labs in FRANCE
4 labs in GERMANY
2 labs in GUATEMALA
11 labs in HONG KONG
1 lab in HUNGARY
6 labs in INDIA
3 labs in INDONESIA
1 lab in ITALY
2 labs in JAPAN
3 labs in KOREA
2 labs in MALAYSIA
5 labs in MEXICO
27 labs in P.R. of CHINA
3 labs in PAKISTAN
2 labs in PHILIPPINES
1 lab in PORTUGAL
1 lab in SINGAPORE
1 lab in SPAIN
1 lab in SRI LANKA
2 labs in SWITZERLAND
1 lab in TAIWAN R.O.C.
2 labs in THAILAND
3 labs in THE NETHERLANDS
4 labs in TURKEY
6 labs in U.S.A.
2 labs in UNITED KINGDOM
2 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:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
2 16 CFR § 1303.1
3 16 CFR § 1303.2
4 ASTM F963-07 Standard Consumer Safety Specification for Toy Safety
5 Horwitz. Journal of AOAC International Vol. 79 No.3 1996
6 P.L. Davies. Fr Z. Anal. Chem. 351 513 (1988)
7 W.J. Conover. Practical; Nonparametric Statistics. J. Wiley&Sons NY, p.302 (1971)
8 ISO 5725 (1986)
9 ISO 5725 parts 1-6 (1994)
10 CPSC-CH-E1002-08
11 CPSC-CH-E1003-09
12 M. Thompson and R. Wood. J. AOAC Int. 76 926 (1993)
13 Analytical Methods Committee Technical brief, No.4 January 2001
14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page 1359-1364, P.J. Lowthian and M. Thompson (see http://www.rsc.org/suppdata/an/b2/b205600n/)