Results of Proficiency Test
Total lead in Paint
March 2010

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: Ing. R.J. Starink
Correctors: Dr. R.G. Visser & Ing. N. Boelhouwer
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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 this international interlaboratory study 112 laboratories in 28 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 and ILAC-G13:2007. 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 of sample #1002 was milled until the particles passed through a 0.5 mm sieve. The dried paint of sample #1001 was used as flakes as it was not possible to mill and sieve it due to the form of the flakes and the fact that this dry paint was very static.
The two dried paint samples, labelled #1001 and #1002 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 #1001-1	785
Sample #1001-2	746
Sample #1001-3	780
Sample #1001-4	794
Sample #1001-5	809

table 1: measured lead contents for homogeneity test of subsamples #1001

	Lead conc. in mg/kg
Sample #1002-1	385
Sample #1002-2	395
Sample #1002-3	382
Sample #1002-4	372
Sample #1002-5	383
Sample #1002-6	381

table 2: measured lead contents for homogeneity test of subsamples #1002

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 #1001	*Lead conc. in mg/kg #1002*
r (observed)	35	21
Reference method	Horwitz	Horwitz
0.3 * R (ref. method)	39	21

table 3: evaluation of the observed repeatabilities of subsamples #1001 and #1002

The calculated repeatabilities are both less than 0.3 times the corresponding reproducibility estimated from the Horwitz equation. Therefore, homogeneity of the subsamples of #1001 and #1002 was assumed.

Approx. 0.5 grams of each of the samples #1001 and #1002 were sent to the participating laboratories on February 18, 2010.

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. 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 3, 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 111 reporting laboratories did report in total 222 numerical results. Observed were 11 statistical outlying results, which is 4.7 % 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 #1001: The total Lead determination on this sample, at a concentration level of 780 mg/kg, may be problematic. Six statistical outliers were observed and the observed reproducibility is, after rejection of the statistical outliers, larger than the strict target reproducibility estimated from the Horwitz equation. Separate evaluation of SPSC-test results and ASTM E1645-test results leads to the same conclusion. The use of the different test methods does not explain for the large spread.

Sample #1002: The total Lead determination on this sample, at a concentration level of 360 mg/kg, may be problematic for a number of laboratories. Five statistical outliers were observed. However, the observed reproducibility, after rejection of the statistical outliers, is almost in agreement with the strict target reproducibility estimated from the Horwitz equation.
Separate evaluation of SPSC-test results leads to the same conclusion. However the spread of the ASTM E1645-test results is larger than the spread of the SPSC-test results.
Although the lead concentration of sample #1002 (356 mg/kg) was well above the limit of 300 mg/kg for substrates in children's products, no less than 5 laboratories did report a test result less than 300 mg/kg.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the strict 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 (Horwitz equation), are compared in the next table.

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Lead #1001	mg/kg	104	778.5	161.5	128.1
Lead #1002	mg/kg	105	355.9	78.0	65.9

table 4: reproducibilities of lead in paint samples #1001 and #1002

From the above table it can be concluded that, without statistical calculations, the group of participating laboratories has 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.

5 DISCUSSION

A large number of different test methods were used. Most often CPSC-CH-E1003-09 was used (46 times) and ASTM E1645 (14 times), followed by 'in house' test methods. Remarkably, most laboratories used both sample 'as received'. Only 17 laboratories did mill (or powder) sample #1001 that clearly consisted of flakes larger than the allowed 0.5 mm. However, as the relative spread for both samples is 21%, 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 very homogeneous. In real world samples this may be different.

Most laboratories did use a 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 all reported test details). Only one laboratory tried to used XRF as test method, but the amount of sample was insufficient to give good test results with this test method.
No significant correlations were found between the reported results and the reported test methods used.

General

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.

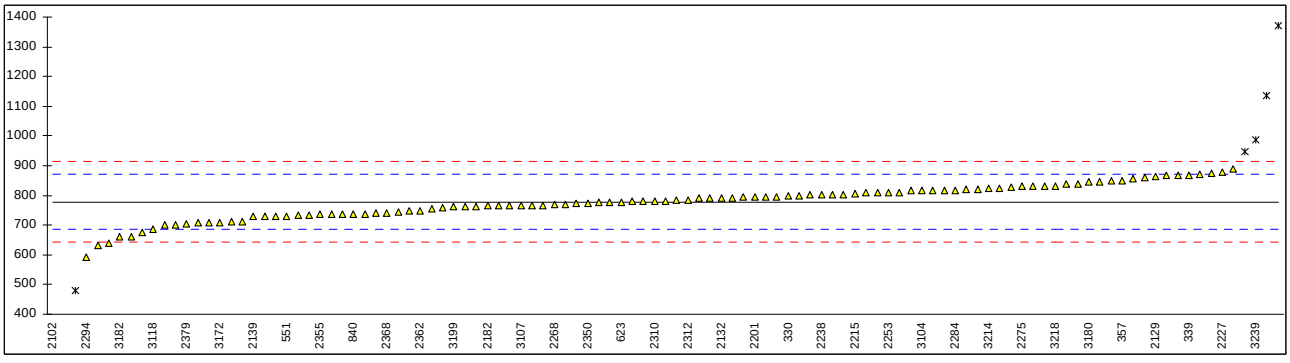
APPENDIX 1

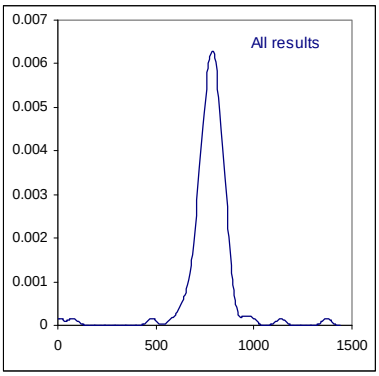
Determination of Total Lead as Pb on sample #1001; results in mg/kg

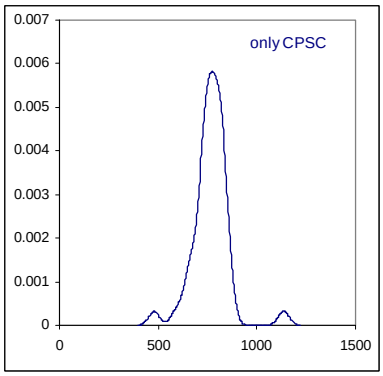
lab	method	value	mark	z(targ)	remarks
110	CPSC-CH-E1003-09	638.7535		-3.06	
330	in house/CPSC	797.9		0.42	
339	in house	868		1.96	
357	in house-528	850		1.56	
551	CPSC-CH-E1003-09	730.76		-1.04	
622	acid digestion	830.8		1.14	
623	CPSC-CH-E1003-09	776.11		-0.05	
632	CPSC-CH-E1003-09	740.7		-0.83	
840	CPSC-CH-E1003-09	736.3		-0.92	
1051	CPSC-CH-E1003-09	827.94		1.08	
1132	ASTM D3335	800.6178		0.48	
1213	ASTM E1645	767.5		-0.24	
2102	in house	8.34	G(0.01)	-16.84	late received revised result 834 mg/kg (z=+1.21)
2117	ISO11885	776		-0.06	
2118	microwave	821.75		0.94	
2129	ICP-MS	863		1.85	
2131	16CFR1303	784.36		0.13	
2132	CPSC-CH-E1003-09	791.80		0.29	
2139	ASTM E1645	728.3		-1.10	
2146	in house	808		0.64	
2152	ICP-AES	794.5		0.35	
2156	CPSC-CH-E1003-09	867.0		1.93	
2160	CPSC-CH-E1003-09	765.6		-0.28	
2165	EPA3052	817.58		0.85	
2170	CPSC-CH-E1003-09	662.028		-2.55	
2172	AOAC974.02	780.7		0.05	
2179	EPA3052	761.8		-0.37	
2182	CPSC-CH-E1003-09	764.4		-0.31	
2184	CPSC-CH-E1003-09	766.3		-0.27	
2185	ASTM E1645 mod	830.48		1.14	
2190	in house	793		0.32	
2196	in house	815.4		0.81	
2201	EPA3052	794.7		0.35	
2215	16CFR1303	806.4		0.61	
2217	IEC62321	890.1		2.44	
2227	ASTM E1645	877.16		2.16	
2228	CPSC-CH-E1003-09	1136.875	G(0.01)	7.84	
2229	CPSC-CH-E1003-09	796		0.38	
2238	EPA3052	801		0.49	
2243	CPSC-CH-E1003-09	707.6		-1.55	
2245	16CFR1303	763.676		-0.32	
2251	EPA3052	857.3051		1.72	
2253	CPSC-CH-E1003-09	810.27		0.69	
2254	microwave	789.6		0.24	
2255	ASTM E1645	632.50		-3.19	
2256	CPSC-CH-E1003-09	728.41		-1.10	
2257		-----		-----	
2258	16CFR1303	781.8		0.07	
2260	CPSC-CH-E1003-09	735.6		-0.94	
2266	CPSC-CH-E1003-09	676	C	-2.24	first reported 1002
2268	CPSC-CH-E1003-09	768.2		-0.23	
2271	ASTM E1645	872.9		2.06	
2275	ASTM E1645	830.0		1.13	
2278	GB/T22788	699.82		-1.72	
2281	16CFR1303	746.30		-0.70	
2283	EPA3050B	846		1.48	
2284	EPA3050B	818.0		0.86	
2289	CPSC-CH-E1003-09	839		1.32	
2293	CPSC-CH-E1003-09	713.250		-1.43	
2294	CPSC-CH-E1003-09	590.4		-4.11	
2310	ASTM E1645	781		0.05	
2311	ASTM E1645	790.4		0.26	
2312	ASTM E1645	785.7		0.16	
2320	in house	708.0		-1.54	
2350	CPSC-CH-E1003-09	774.8		-0.08	
2353	EPA3051	770		-0.19	
2355	EPA3052	735.4		-0.94	
2357	ASTM E1645	734.38		-0.97	
2359	CPSC-CH-E1003-09	733.8		-0.98	
2362	CPSC-CH-E1003-09	748		-0.67	
2363	CPSC-CH-E1003-09	729.9		-1.06	
2365	EPA3052	710.6		-1.49	
2366	CPSC-CH-E1003-09	771.4		-0.16	
2368	ASTM E1645	741.1		-0.82	
2369	CPSC-CH-E1003-09	736.8		-0.91	

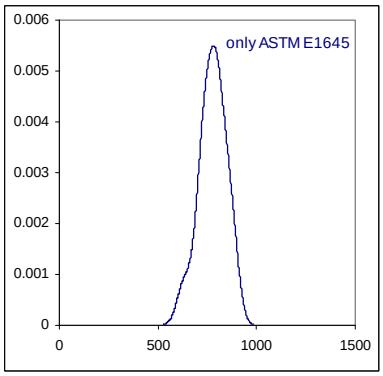
2370	CPSC-CH-E1003-09	780		0.03	
2372	EPA3052	754.1		-0.53	
2375	in house	735.5		-0.94	
2379	ASTM E1645	704.50		-1.62	
2380	CPSC-CH-E1003-09	767.292		-0.25	
2385	ICP-MS	948.0	DG(0.05)	3.71	
3100	CPSC-CH-E1003-09	848.0		1.52	
3104	CPSC-CH-E1003-09	817.5		0.85	
3107	16CFR1303	767		-0.25	
3116	CPSC-CH-E1003-09	802.95		0.53	
3117	CPSC-CH-E1003-09	821.59		0.94	
3118	CPSC-CH-E1003-09	687.8		-1.98	
3124	EPA3052	757		-0.47	
3151	in house	870		2.00	
3153	CPSC-CH-E1003-09	811.0		0.71	
3154	in house	742.5		-0.79	
3159	CPSC-CH-E1003-09	859		1.76	reported 0.0859%
3163	XRF	-----		----	too little sample for XRF (found only 78.1 mg/kg)
3166	ICP-MS	866		1.91	
3167	CPSC-CH-E1003-09	480.1	G(0.01)	-6.53	
3169	CPSC-CH-E1003-09	775.502		-0.07	
3172	in house	709.1		-1.52	
3176	16CFR1303	1371	G(0.01)	12.96	
3180		845		1.45	
3182	CPSC-CH-E1003-09	660.65		-2.58	
3185	CPSC-CH-E1003-09	836.6		1.27	
3190	ASTM E1645	808		0.64	
3199	in house	761.7		-0.37	
3200	microwave	701.13		-1.69	
3209	EPA3052	795.2		0.36	
3210	CPSC-CH-E1003-09	825		1.02	
3214	CPSC-CH-E1003-09	823.81		0.99	
3218	EPA3052	832.2		1.17	
3222	CPSC-CH-E1003-09	818.0		0.86	
3228	microwave	803.7		0.55	
3239	in house	988.692	DG(0.05)	4.60	
3248	CPSC-CH-E1003-09	800		0.47	

			Only CPSC-results	Only E1645-results
	normality	OK	OK	OK
	n	104	44	14
	outliers	6	2	0
	mean (n)	778.54	765.04	777.42
	st.dev. (n)	57.680	61.283	66.698
	R(calc.)	161.50	(=21%)	171.59	186.75
	R(Horwitz)	128.05		126.16	127.89







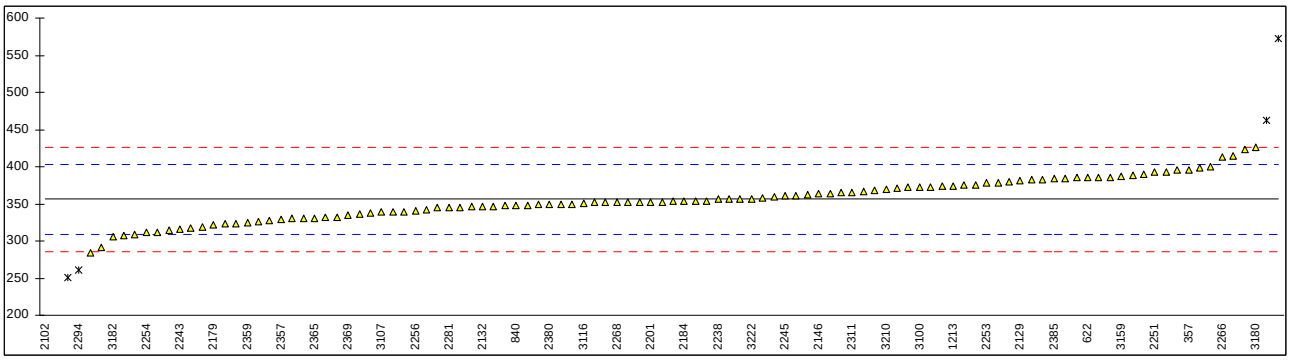


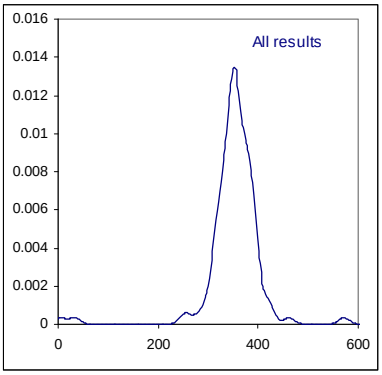
Determination of Total Lead as Pb on sample #1002; results in mg/kg

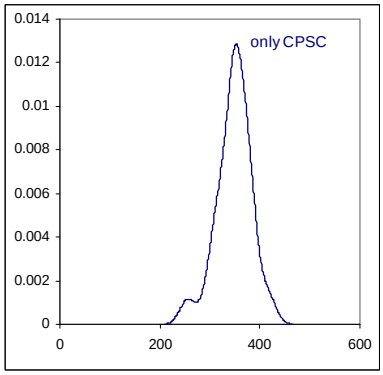
lab	method	value	mark	z(targ)	remarks
110	CPSC-CH-E1003-09	291.0685		-2.75	
330	in house/CPSC	339.1		-0.71	
339	in house	383		1.15	
357	in house-528	396		1.71	
551	CPSC-CH-E1003-09	323.61		-1.37	
622	acid digestion	385.2		1.25	
623	CPSC-CH-E1003-09	345.82		-0.43	
632	CPSC-CH-E1003-09	326.4		-1.25	
840	CPSC-CH-E1003-09	348.2		-0.33	
1051	CPSC-CH-E1003-09	378.71		0.97	
1132	ASTM D3335	395.1595		1.67	
1213	ASTM E1645	374.0		0.77	
2102	in house	4.10	G(0.01)	-14.96	late received revised result 410 mg/kg (z=+2.30)
2117	ISO11885	367		0.47	
2118	microwave	393.38		1.60	
2129	ICP-MS	381		1.07	
2131	16CFR1302	349.89		-0.25	
2132	CPSC-CH-E1003-09	346.34		-0.40	
2139	ASTM E1645	317.6		-1.63	
2146	in house	364		0.35	
2152	ICP-AES	330.51		-1.08	
2156	CPSC-CH-E1003-09	373.7		0.76	
2160	CPSC-CH-E1003-09	348.5		-0.31	
2165	EPA3052	368.21		0.53	
2170	CPSC-CH-E1003-09	306.703		-2.09	
2172	AOAC974.02	350.0		-0.25	
2179	EPA3052	322.1		-1.44	
2182	CPSC-CH-E1003-09	360.1		0.18	
2184	CPSC-CH-E1003-09	353.4		-0.10	
2185	ASTM E1645 mod	384.16		1.20	
2190	in house	357		0.05	
2196	in house	351.9		-0.17	
2201	EPA3052	352.6		-0.14	
2215	16CFR1303	362.7		0.29	
2217	IEC62321	414.1		2.48	
2227	ASTM E1645	390.19		1.46	
2228	CPSC-CH-E1003-09	423.125		2.86	
2229	CPSC-CH-E1003-09	386		1.28	
2238	EPA3052	356		0.01	
2243	CPSC-CH-E1003-09	315.9		-1.70	
2245	16CFR1303	360.818		0.21	
2251	EPA3052	392.9036		1.58	
2253	CPSC-CH-E1003-09	378.21		0.95	
2254	microwave	311.5		-1.89	
2255	ASTM E1645	284.25		-3.04	
2256	CPSC-CH-E1003-09	340.93		-0.63	
2257		-----		-----	
2258	16CFR1303	335.7		-0.86	
2260	CPSC-CH-E1003-09	354.2		-0.07	
2266	CPSC-CH-E1003-09	413		2.43	
2268	CPSC-CH-E1003-09	351.6		-0.18	
2271	ASTM E1645	398.3		1.80	
2275	ASTM E1645	388.3		1.38	
2278	GB/T22788	331.94		-1.02	
2281	16CFR1303	345.24		-0.45	
2283	EPA3050B	383		1.15	
2284	EPA3050B	385.3		1.25	
2289	CPSC-CH-E1003-09	379		0.98	
2293	CPSC-CH-E1003-09	332.067		-1.01	
2294	CPSC-CH-E1003-09	261.2	DG(0.05)	-4.03	
2310	ASTM E1645	342		-0.59	
2311	ASTM E1645	365.2		0.40	
2312	ASTM E1645	345.5		-0.44	
2320	in house	349.5		-0.27	
2350	CPSC-CH-E1003-09	345.2		-0.45	
2353	EPA3051	348		-0.33	
2355	EPA3052	329.8		-1.11	
2357	ASTM E1645	329.30		-1.13	
2359	CPSC-CH-E1003-09	324.5		-1.33	
2362	CPSC-CH-E1003-09	347		-0.38	
2363	CPSC-CH-E1003-09	327.0		-1.23	
2365	EPA3052	330.7		-1.07	
2366	CPSC-CH-E1003-09	352.8		-0.13	
2368	ASTM E1645	364.2		0.35	
2369	CPSC-CH-E1003-09	334.9		-0.89	

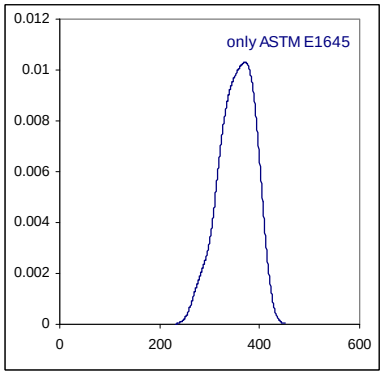
2370	CPSC-CH-E1003-09	354		-0.08	
2372	EPA3052	339.1		-0.71	
2375	in house	371.8		0.68	
2379	ASTM E1645	323.35		-1.38	
2380	CPSC-CH-E1003-09	349.566		-0.27	
2385	ICP-MS	383.5		1.18	
3100	CPSC-CH-E1003-09	372.8		0.72	
3104	CPSC-CH-E1003-09	399.5		1.86	
3107	16CFR1303	339		-0.72	
3116	CPSC-CH-E1003-09	351.15		-0.20	
3117	CPSC-CH-E1003-09	375.63		0.84	
3118	CPSC-CH-E1003-09	308.3		-2.02	
3124	EPA3052	365		0.39	
3151	in house	371		0.64	
3153	CPSC-CH-E1003-09	353.0		-0.12	
3154	in house	351.6		-0.18	
3159	CPSC-CH-E1003-09	387		1.32	reported 0.0387%
3163	XRF	-----		----	too little sample for XRF (found only 33.5 mg/kg)
3166	ICP-MS	385		1.24	
3167	CPSC-CH-E1003-09	250.5	DG(0.05)	-4.48	
3169	CPSC-CH-E1003-09	361.352		0.23	
3172	in house	314.5		-1.76	
3176	16CFR1303	572	G(0.01)	9.19	
3180		426		2.98	
3182	CPSC-CH-E1003-09	306.38		-2.10	
3185	CPSC-CH-E1003-09	372.8		0.72	
3190	ASTM E1645	352		-0.16	
3199	in house	338.2		-0.75	
3200	microwave	318.63		-1.58	
3209	EPA3052	351.5		-0.19	
3210	CPSC-CH-E1003-09	370		0.60	
3214	CPSC-CH-E1003-09	375.13		0.82	
3218	EPA3052	357.9		0.09	
3222	CPSC-CH-E1003-09	357.2		0.06	
3228	microwave	357.0		0.05	
3239	in house	462.583	G(0.05)	4.54	
3248	CPSC-CH-E1003-09	312		-1.86	

			Only CPSC-results	Only E1645-results
	normality	OK	OK	OK
	n	105	44	14
	outliers	5	2	0
	mean (n)	355.86	352.59	354.17
	st.dev. (n)	27.865	28.516	32.755
	R(calc.)	78.02	(=21%)	79.84	91.71
	R(Horwitz)	65.85		65.33	65.58









APPENDIX 2

Analytical details as used by the participants

Lab	Digestion technique	Pretreatment	Digestion liquid	s/l ratio	Detection
110	acid digestion	as received	HNO3	100mg/25mL	AAS
330	microwave	as received	HNO3	100mg/25ml	ICP-OES
339	--	--	--	--	--
357	microwave	as received	HNO3	200mg/10ml	ICP
551	microwave	as received	HNO3	200mg/25mL	ICP-OES
622	acid digestion	powdered	HNO3	250mg/50mL	AAS
623	microwave	as received	HNO3/H2O2	2mg/1mL*	ICP-MS
632	microwave	as received	HNO3	100mg/10mL	ICP-OES
840	microwave	as received	HNO3	100mg/10mL	ICP-OES
1051	acid digestion	as received	HNO3/H2O2	300mg/25mL	ICP
1132	Ashing	as received	HNO3	100mg/100mL	AAS
1213	microwave	powdered	HCl:HNO3=3:1	250mg/8mL	ICP-MS
2102	microwave	as received	HNO3	100mg/5mL	ICP-MS
2117	microwave	as received	HNO3	100mg/10mL	ICP
2118	microwave	as received	HNO3/H2O2	100mg/100mL	ICP-OES
2129	microwave	as received	HNO3	100mg/50mL	ICP-MS
2131	microwave	as received	HNO3	100mg/10mL	ICP-MS
2132	microwave	as received	HNO3	100mg/5mL	AAS
2139	microwave	as received	HNO3	100mg/50mL	ICP
2146	acid digestion	as received	HNO3:H2O2	100mg/25mL	ICP
2152	microwave	as received	HNO3	0.3mg/10mL*	ICP-AES
2156	acid digestion	as received	HNO3	25mg/25mL	ICP-OES
2160	microwave	#1001: milled; #1002: as received	HNO3/H2O2	100mg/50mL	AAS
2165	microwave	as received	HNO3	100mg/10mL	AAS
2170	microwave	as received	HNO3	100mg/10mL	ICP
2172	hot plate	as received	HNO3	100mg/25mL	ICP-OES
2179	microwave	as received	HNO3:HF:H2O2=6:1:2	100mg/50mL	ICP
2182	microwave	as received	HNO3	100mg/5mL	ICP
2184	microwave	as received	HNO3	100mg/10mL	ICP
2185	microwave	as received	HNO3/H2O2	200mg/20mL	AAS
2190	microwave	as received	HNO3	200mg/50mL	ICP
2196	acid digestion	powdered	HNO3, H2O2	100mg/25mL	AAS
2201	microwave	milled	HNO3	100mg/8mL	ICP
2215	acid digestion	as received	HNO3	200mg/10mL	ICP-OES
2217	microwave	as received	HNO3/H2O2	150mg/6mL	AAS
2227	hot plate	as received	HNO3	100mg/10mL	ICP
2228	hot plate	as received	HNO3	80mg/10mL	ICP-MS
2229	hot plate	milled	HNO3	100mg/15mL	AAS
2238	microwave	as received	HNO3	1mg/1mL	AAS
2243	microwave	as received	HNO3	100mg/5mL	ICP-OES
2245	microwave	powdered	HNO3: H2O2	100mg/10mL	ICP-OES
2251	microwave	as received	HNO3/HF/H2O2	0.1mg/100mL*	AAS
2253	microwave	as received	HNO3	100mg/10mL	ICP
2254	microwave	powdered	HCl/HNO3/H2O2	200mg/25mL	ICP
2255	acid digestion	as received	HNO3		ICP
2256	acid digestion	as received	HNO3	100mg/25mL	ICP-OES
2257					
2258	acid digestion	powdered	HNO3		ICP
2260	microwave	as received	HNO3	100mg/10mL	ICP
2266	acid digestion	dryed	HNO3	200mg/5mL	AAS
2268	microwave		HNO3	200mg/50mL	ICP-OES
2271	acid digestion	as received	HCl/HNO3/H2O2	100mg/100mL	ICP-OES
2275	acid digestion	as received	HNO3: H2O2	10mg/100mL	ICP
2278	microwave	as received	HNO3: H2O2	100mg/20mL	ICP

2281	microwave	powdered	HNO3: H2O2	100mg/10mL	ICP-OES
2283	acid digestion	as received	HNO3/H2O2/HCl	300mg/50mL	AAS
2284	acid digestion	as received	HNO3/H2O2	100mg/50mL	ICP-OES
2289	microwave	as received	HNO3	85mg/6ml	ICP
2293	acid digestion	scraped	HNO3	50-150mg/6mL	ICP
2294	microwave	as received	HNO3/HCl	100mg/10mL	ICP
2310	microwave	as received	HNO3	100mg/10mL	AAS
2311	microwave	as received	HNO3	100mg/8ml	AAS
2312	microwave	as received	HNO3	0.5mg/1mL*	ICP-OES
2320	microwave	as received	HNO3	100mg/10mL	AAS
2350	microwave	powdered	HNO3+H2O2	100mg/25mL	ICP-OES
2353	microwave	as received	HNO3	100mg/10mL	ICP-OES
2355	microwave	as received	HNO3/HF	100mg/25mL	ICP
2357	microwave	milled	HNO3	100mg/25mL	ICP
2359	acid digestion	as received	HNO3	100mg/25mL	AAS
2362	microwave	as received	HNO3	100mg/10mL	ICP-OES
2363	acid digestion	as received	HNO3/H2O2	100mg/50mL	ICP-OES
2365	microwave	as received	HNO3/HF	0.1mg/25mL*	ICP-OES
2366	acid digestion	as received	HNO3	100mg/10mL	ICP-OES
2368	acid digestion	milled	HNO3	250mg/50mL	AAS
2369	microwave	as received	HNO3+H2O2	100mg/25mL	ICP
2370	microwave	as received	HNO3	100mg/5ml	ICP-AES
2372	acid digestion	as received	HCl/HNO3/HF	200mg/25mL	ICP-AES
2375	microwave	as received	HNO3	12.5mg/25mL	ICP-MS
2379	microwave	powdered	HNO3	100mg/25mL	AAS
2380	acid digestion	as received	HNO3	50mg/10mL	ICP-OES
2385	microwave	as received	HNO3/H2O2	200mg/6mL	ICP-OES
3100	microwave	as received	HNO3	100mg/50mL	ICP
3104	microwave	as received	HNO3	1mg/25mL*	AAS
3107	microwave	as received	HNO3	50mg/25mL	ICP
3116	microwave	as received	HNO3	100mg/25mL	ICP
3117	microwave	as received	HNO3	30mg/10mL	ICP-MS
3118	microwave	as received	HNO3	100mg/10mL	ICP-OES
3124	microwave	as received	HNO3/HF	250mg/11mL	ICP-MS
3151	acid digestion	as received	HNO3	100mg/20mL	ICP-MS
3153	microwave	as received	HNO3	100mg/25mL	ICP
3154	microwave	as received	HNO3/H2O2	100mg/8mL	ICP
3159	microwave	as received	HNO3	100mg/25mL	ICP-OES
3163					
3166	microwave	as received	HNO3	100mg/50mL	ICP-MS
3167	microwave	as received	HNO3	0.1/25ml *	ICP
3169	microwave	as received	HNO3	100mg/5mL	ICP-OES
3172	microwave	as received	HNO3	40mg/8mL	ICP
3176	microwave	as received	H2SO4/HNO3	100mg/10mL	ICP-MS
3180	microwave		HNO3/HCl/H2O2	200mg/50mL	AAS
3182	microwave	as received	HNO3	100mg/25mL	ICP
3185	microwave	powdered	HNO3	0.1mg/25mL *	ICP
3190	microwave	as received	HNO3/HF	200mg/50mL	ICP
3199	hot plate	as received	HNO3	100mg/10mL	ICP
3200	microwave	as received	HNO3/HCl	100mg/15mL	AAS
3209	microwave	as received	HNO3	100mg/25mL	AAS
3210	microwave		HNO3/HCl	0.1mg/50mL *	ICO/AES
3214	microwave	powdered	HNO3	100mg/50mL	ICP-OES
3218	microwave	milled	HNO3	200mg/10mL	ICP-OES
3222	microwave	as received	HNO3	200mg/25mL	AAS
3228	microwave	as received	HNO3	100mg/50ml	ICP-OES
3239	acid digestion	as received	HNO3/HCl	100mg/25mL	ICP
3248	microwave	as received	HNO3	150mg/5mL	ICP-OES

* s/l ratio was probably was reported in g/mL in stead of in mg/mL

APPENDIX 3

Number of participants in alphabetic country order:

2 labs in	BANGLADESH
1 lab in	BELGIUM
2 labs in	BRASIL
1 lab in	DENMARK
2 labs in	FINLAND
5 labs in	FRANCE
5 labs in	GERMANY
2 labs in	GUATEMALA
14 labs in	HONG KONG
1 lab in	HUNGARY
3 labs in	INDIA
3 labs in	INDONESIA
2 labs in	ITALY
2 labs in	KOREA
1 lab in	MALAYSIA
2 labs in	MEXICO
38 labs in	P.R. of CHINA
1 lab in	PHILIPPINES
2 labs in	SINGAPORE
1 lab in	SRI LANKA
2 labs in	SWITZERLAND
3 labs in	TAIWAN R.O.C.
3 labs in	THAILAND
2 labs in	THE NETHERLANDS
3 labs in	TURKEY
5 labs in	U.S.A.
1 lab 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 i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
2 16 CFR § 1303.1
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6 P.L. Davies. Fr Z. Anal. Chem. 351. 513. (1988)
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10 CPSC-CH-E1002-08
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12 M. Thompson and R. Wood. J. AOAC Int. 76. 926. (1993)
13 Analytical Methods Committee Technical brief, No4 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/).