

**Results of Proficiency Test
Total lead in Paint
April 2014**

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 2014 interlaboratory study on total lead in paint 134 laboratories in 34 different countries participated. See appendix 2 for the number of participants per country. In this report the results of the 2014 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies in Spijkenisse was the organiser of this proficiency test. It was decided to use in this proficiency test 2 different paint samples with concentrations near the 200 and 170 mg/kg limits. The fit-for-use and homogeneity testing was subcontracted.

The participants were asked to report the analytical results with one extra figure using the indicated units on the report form. These results with an extra figure are preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). 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 organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded via the FAQ page of the iis website <http://www.iisnl.com>.

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

Two different paint samples were used in this proficiency test. Sample #14050 was made from an organic based paint and sample #14051 was made from a water based paint. To sample #14050 very fine lead oxide was added and to sample #14051 lead nitrate dissolved in water 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 #14050 and #14051 were respectively divided over 172 and 193 subsamples of 0.5 gram each. The sub samples, labelled #14050 and labelled #14051 were tested for homogeneity on 8 randomly selected samples each. The analytical testing was performed by a subcontracted laboratory. See the following tables for the homogeneity test results.

	Lead conc. in mg/kg
Sample #14050-1	192
Sample #14050-2	186
Sample #14050-3	185
Sample #14050-4	188
Sample #14050-5	189
Sample #14050-6	188
Sample #14050-7	184
Sample #14050-8	189

table 1: homogeneity test results of subsamples #14050

	Lead conc. in mg/kg
Sample #14051-1	171
Sample #14051-2	176
Sample #14051-3	174
Sample #14051-4	174
Sample #14051-5	181
Sample #14051-6	178
Sample #14051-7	176
Sample #14051-8	182

table 2 : homogeneity test results of subsamples #14051

From the homogeneity test results of table 1 and table 2, 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:

	#14050 mg/kg	#14051 mg/kg
r (observed)	7	10
Reference method	Horwitz	Horwitz
0.3 * R (ref. method)	11	11

table 3: repeatabilities of subsamples #14050 and subsamples #14051

The calculated repeatabilities for samples #14050 and #14051 are both in agreement with 0.3 times the estimated target reproducibilities, calculated using the Horwitz equation. Therefore, homogeneity of the subsamples #14050 and #14051 was assumed.

Approx. 0.5 grams of each of the samples #14050 and #14051 were sent to the participating laboratories on April 9, 2014.

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, on which the units were prescribed and a letter of instructions were sent together with each set of samples.

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 3.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3)

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.

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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 (1986 and 1994, lit.8 and 9) the original results per determination were submitted subsequently to Dixon's, Grubbs' and Rosner 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 and by $R(0.01)$ for the Rosner General ESD test (ref. 15). 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 General ESD test (ref. 15). 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 3, nos.13-14). Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

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. These 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 whether the reported test result is fit-for-use.

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:

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

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

During the execution of this proficiency test some reporting problems occurred. Of the 134 participants, 21 participants reported results after the final reporting date and two laboratories reported no results at all.

Finally, the 132 reporting laboratories did report in total 264 numerical results. Observed were 10 statistically outlying results, which is 3.8% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not normal Gaussian distribution was found for the lead determination in both samples (#14050 and #14051). Therefore, the statistical evaluation for the determinations 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 an 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 #14050: The total lead determination on this sample, at a concentration level of 197 mg/kg, may be problematic for a number of laboratories. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated reproducibility calculated using the Horwitz equation. When the 90 reported CPSC test results are evaluated separately, the calculated reproducibility is the same as the calculated reproducibility for all data.

Sample #14051: The total lead determination on this sample, at a concentration level of 170 mg/kg, may be problematic for a number of laboratories. Five statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers is in good agreement with the estimated reproducibility calculated using the Horwitz equation. When the 89 reported CPSC test results are evaluated separately, the calculated reproducibility is smaller than the calculated reproducibility of all data.

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.

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R (target)</i>
Total Lead in #14050	mg/kg	127	197.5	34.9	39.9
Total Lead in #14051	mg/kg	127	170.2	36.1	35.2

table 4: reproducibilities of lead in paint samples #14050 and #14051

From the above table it can be concluded, without statistical calculations, that the participating laboratories have no 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 EVALUATION OF THE PROFICIENCY TEST OF APRIL 2014 WITH PREVIOUS PTS

	<i>April 2014</i>	<i>April 2013</i>	<i>February 2012</i>	<i>February 2011</i>
Number of reporting labs	132	139	110	86
Number of results reported	264	276	215	172
Number of statistical outliers	10	6	9	5
Percentage outliers	3.8%	2.2%	4.2%	2.8%

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 is summarized in table 6.

PARAMETER	<i>April 2014</i>	<i>April 2013</i>	<i>February 2012</i>	<i>February 2011</i>	<i>February 2010</i>	<i>February 2009</i>
30-300 mg Pb/kg	6 - 8%	10%	10%	9%	n.e.	8%
300-900 mg Pb/kg	n.e.	n.e.	n.e.	8%	7 - 8%	7%

Table 6: comparison of the uncertainties (in %) in the previous and present PT

5 DISCUSSION

A large number of different test methods were used. The CPSC-CH-E1003-09 method was used by 93 laboratories and other versions of CPSC were used 1 time. Twenty-three laboratories reported to have used 'in house' methods. ASTM E1645 was used 4 times. Other laboratories reported to have used ASTM F963, EPA3051A, EPA3052, ISO17294-2, GB/T 22788 and GB24613. These methods were all mentioned once.

When the CPSC test results are evaluated separately then the calculated reproducibilities are somewhat smaller than the precision of all data for both samples. This was to be expected as this selected group of laboratories followed the same analytical procedure.

To prepare sample #14050 finely powdered lead oxide was added to an organic based paint and to sample #14051 a solution of lead nitrate in water was added to a water based paint. As the observed spreads in the reported lead concentrations do not differ significantly, the type of paint and the type of lead salt obviously have no significant effect on the quality of the test results.

APPENDIX 1

Determination of Total Lead as Pb on sample #14050; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	in house	197.84		0.03	2464	CPSC-CH-E1003-09	218.5		1.48
213	CPSC-CH-E1003-09.1	183.529		-0.98	2465	ASTM E1645	201.2		0.26
311	in house	206.5		0.63	2471	CPSC-CH-E1003-09.1	198.94		0.10
330	in house	204.7		0.51	2476	CPSC-CH-E1003-09.1	186.2		-0.79
551	in house	208.25		0.76	2480	in house	202.9	C	0.38
622	in house	187.3261		-0.71	2482	CPSC-CH-E1003-09.1	218.925		1.51
632	in house	183.83		-0.96	2488	CPSC-CH-E1003-09	194.57		-0.20
1213	CPSC-CH-E1003-09.1	211.11		0.96	2489	CPSC-CH-E1003-09.1	191.48		-0.42
2102	in house	201.623		0.29	2492	in house	205.7		0.58
2108	CPSC	203.6		0.43	2495	CPSC-CH-E1003-09	210.1		0.89
2115		-----		-----	2500	CPSC-CH-E1003-09.1	198.7		0.09
2118	in house	191.52		-0.42	2503	CPSC-CH-E1003-09.1	217.8		1.43
2129	ISO17294	196.6		-0.06	2511	CPSC-CH-E1003-09	182.55		-1.05
2131	in house	178.54		-1.33	2514	CPSC-CH-E1003-09.1	196.95		-0.04
2132	CPSC-CH-E1003-09.1	195.69		-0.12	2522	CPSC-CH-E1003-09	198.3		0.06
2135	§LFGB K 84.00-29	182.2		-1.07	2529	CPSC-CH-E1003-09	194.29		-0.22
2137	CPSC-CH-E1003-09	203.6		0.43	2532	CPSC-CH-E1003-09.1	193.5		-0.28
2139	CPSC-CH-E1003-09.1	212		1.02	2545	GB/T 22788	202.3		0.34
2156	CPSC-CH-E1003-09	158.95		-2.70	2548	CPSC-CH-E1003-09.1	202.2		0.33
2170	ASTM E1645	190.0		-0.52	2553	CPSD AN-0001	188.950		-0.60
2172	CPSC-CH-E1003-09.1	197.6		0.01	2564		200.447		0.21
2182	CPSC-CH-E1003-09.1	207.67		0.72	2566	CPSC-CH-E1003-09.1	198.25		0.06
2184	CPSC-CH-E1003-09.1	197.6		0.01	2572	CPSC-CH-E1003-09.1	195.4		-0.14
2190		182.85		-1.02	2574	in house	18.46	R(0.01)	-12.55
2201	CPSC-CH-E1003	204.8		0.52	2581	in house	202.8315		0.38
2225	CPSC-CH-E1003-09.1	209.4		0.84	2582	CPSC-CH-E1003-09.1	179.2		-1.28
2226	EPA6010	207.0		0.67	2590	EN14602	172.8		-1.73
2229	CPSC-CH-E1003-09.1	183.40		-0.99	2591	in house	156.57		-2.87
2232	CPSC-CH-E1003-09.1	190.1		-0.52	2597	GB/T 22788	184.8	C	-0.89
2234	CPSC-CH-E1003-09.1	203.3		0.41	2613	CPSC-CH-E1003-09.1	206.07		0.60
2236	CPSC-CH-E1003-09.1	197.6		0.01	2614	CPSC-CH-E1003-09.1	200.24		0.20
2238	CPSC-CH-E1003-09.1	196		-0.10	2625	CPSC-CH-E1003-09.1	237.05		2.78
2245	CPSC-CH-E1003-09	201.9		0.31	3100	CPSC-CH-E1003-09.1	200.18		0.19
2246	CPSC-CH-E1003-09	212		1.02	3107	CPSC-CH-E1003-09	181.7		-1.10
2247	CPSC-CH-E1003-09.1	196.81		-0.05	3110	CPSC-CH-E1003-09.1	197.64		0.01
2254	CPSC-CH-E1003-09.1	269.5	R(0.01)	5.05	3116	CPSC-CH-E1003-09.1	202.6		0.36
2255	CPSC-CH-E1003-09.1	198.0		0.04	3118	CPSC-CH-E1003-09	185.9		-0.81
2256	CPSC-CH-E1003-09.1	197.25		-0.01	3122	CPSC-CH-E1003-09.1	212		1.02
2258	CPSC-CH-E1003-09	114.85	C,R(0.01)	-5.79	3124	EPA3052	221		1.65
2268	CPSC-CH-E1003-09.1	208.1		0.75	3146	CPSC-CH-E1003-09.1	207		0.67
2269	in house	202.5		0.35	3151	in house	199.925		0.17
2277	in house	215.58		1.27	3160	CPSC-CH-E1003-09.1	191.07		-0.45
2286	CPSC-CH-E1003-09.1	193.8		-0.26	3163	in house	210		0.88
2287	ASTM E1645	193.0		-0.31	3167	CPSC-CH-E1003-09.3	190.9		-0.46
2289	CPSC-CH-E1003-09.1	205.4		0.56	3172	CPSC-CH-E1003-09	168.5		-2.03
2290	CPSC-CH-E1003-09.1	195.5		-0.14	3176	CPSC-CH-E1003-09.1	194.99		-0.17
2293	CPSC-CH-E1003-09.1	209.53		0.85	3180	EN1122	182.90		-1.02
2295	CPSC-CH-E1003-09.1	201.35		0.27	3182	CPSC-CH-E1003-09.1	185.68		-0.83
2296	in house	182.468		-1.05	3185	CPSC-CH-E1003-09.1	206.4		0.63
2301	CPSC-CH-E1003-09	177.25		-1.42	3190	CPSC-CH-E1003	202		0.32
2366	CPSC-CH-E1003-09.1	194.6		-0.20	3197	CPSC-CH-E1003-09.1	201.78		0.30
2367	CPSC-CH-E1003-09.1	203.4		0.42	3199	in house	194.3	C	-0.22
2370	CPSC-CH-E1003-09.1	180		-1.22	3210	CPSC-CH-E1003-09	203		0.39
2372	CPSC-CH-E1003-09	195.12		-0.16	3214	CPSC-CH-E1003-09.1	216.2		1.31
2375	CPSC-CH-E1003-09.1	204		0.46	3218	CPSC-CH-E1003-09	202.0		0.32
2380	CPSC-CH-E1003-09.1	219.32		1.53	3220	16CFR1303	150.5	C,R(0.05)	-3.29
2385	in house	203		0.39	3228	CPSC-CH-E1003-09.1	195.3		-0.15
2390	CPSC-CH-E1003-09.1	171.56		-1.82	3237	CPSC-CH-E1003-09.1	197.48		0.00
2391	CPSC-CH-E1003-09.1	199.86		0.17	3242	CPSC-CH-E1003-09.1	215.1		1.24
2409	ASTM F963	193.60		-0.27	3248	CPSC-CH-E1003-09.1	204		0.46
2410	CPSC-CH-E1003-09.1	195.74		-0.12	8005	in house	198.3		0.06
2413	CPSC-CH-E1003-09.1	164.4		-2.32	8010	CPSC-CH-E1003-09	209.0262		0.81
2426	CPSC-CH-E1003-09.1	184.9		-0.88					
2429	CPSC-CH-E1003-09	202.4		0.35					
2431	in house	198.0159		0.04					
2433	ASTM E1645	205.12		0.54					
2441	CPSC-CH-E1003-09	196.3		-0.08					
2450	CPSC-CH-E1003-09.1	198.7		0.09					
2452	CPSC-CH-E1001-08.1	118.36	R(0.01)	-5.55					
2453	CPSC-CH-E1003-09.1	206.7		0.65					
2459	CPSC-CH-E1003-09.1	180.38		-1.20					
2460		-----		-----					

Only CPSC-CH-E1003-09

normality	suspect	suspect
n	127	90
outliers	5	3
mean (n)	197.456	198.226
st.dev. (n)	12.4493	12.3672
R(calc.)	34.858	34.628
R(Horwitz)	39.925	40.057

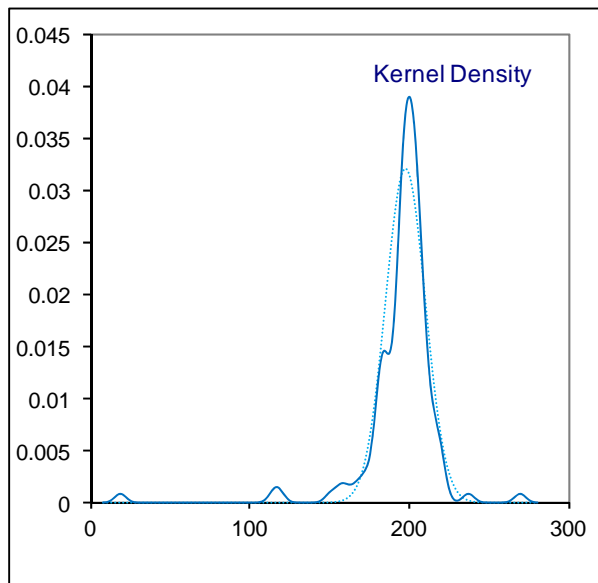
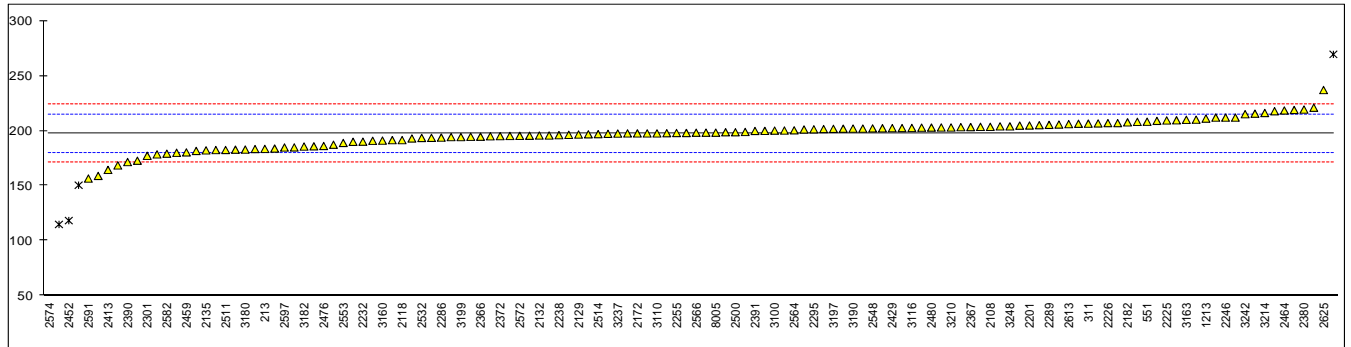
Lab 2258: first reported 411.183

Lab 2480: first reported 249.9

Lab 2597: first reported 160.7

Lab 3199: first reported 234.9

Lab 3220: first reported 286.0



Determination of Total Lead as Pb on sample #14051; results in mg/kg

lab	method	value	mark	z(targ)	lab	method	value	mark	z(targ)
110	in house	154.43		-1.25	2464	CPSC-CH-E1003-09	152.9		-1.37
213	CPSC-CH-E1003-09.1	178.2	C	0.64	2465	ASTM E1645	179.2		0.72
311	in house	188.6		1.47	2471	CPSC-CH-E1003-09.1	169.85		-0.03
330	in house	185.8		1.24	2476	CPSC-CH-E1003-09.1	159.6		-0.84
551	in house	182	C	0.94	2480	in house	199.3		2.32
622	in house	164.6429		-0.44	2482	CPSC-CH-E1003-09.1	185.253		1.20
632	in house	159.03		-0.89	2488	CPSC-CH-E1003-09	178.87		0.69
1213	CPSC-CH-E1003-09.1	174.18		0.32	2489	CPSC-CH-E1003-09.1	158.47		-0.93
2102	in house	148.962		-1.69	2492	in house	176.3		0.49
2108	CPSC	174.4		0.34	2495	CPSC-CH-E1003-09	181.0		0.86
2115		-----		-----	2500	CPSC-CH-E1003-09.1	170.2		0.00
2118	in house	165.37		-0.38	2503	CPSC-CH-E1003-09.1	177.5		0.58
2129	ISO17294	165.2		-0.40	2511	CPSC-CH-E1003-09	166.40		-0.30
2131	in house	165.175		-0.40	2514	CPSC-CH-E1003-09.1	175.0		0.38
2132	CPSC-CH-E1003-09.1	173.41		0.26	2522	CPSC-CH-E1003-09	167.5		-0.21
2135	§LFGB K 84.00-29	156.2		-1.11	2529	CPSC-CH-E1003-09	167.22		-0.24
2137	CPSC-CH-E1003-09	172.4		0.18	2532	CPSC-CH-E1003-09.1	159.5		-0.85
2139	CPSC-CH-E1003-09.1	183		1.02	2545	GB/T 22788	177.3		0.57
2156	CPSC-CH-E1003-09	140.65		-2.35	2548	CPSC-CH-E1003-09.1	176.8		0.53
2170	ASTM E1645	166.4		-0.30	2553	CPSD AN-0001	170.972		0.06
2172	CPSC-CH-E1003-09.1	170.2		0.00	2564		171.179		0.08
2182	CPSC-CH-E1003-09.1	187.29		1.36	2566	CPSC-CH-E1003-09.1	175.4		0.42
2184	CPSC-CH-E1003-09.1	174.8		0.37	2572	CPSC-CH-E1003-09.1	160.2		-0.79
2190		149.70		-1.63	2574	in house	81.80	R(0.01)	-7.03
2201	CPSC-CH-E1003	169.5		-0.05	2581	in house	175.104		0.39
2225	CPSC-CH-E1003-09.1	168.6		-0.13	2582	CPSC-CH-E1003-09.1	155.7		-1.15
2226	EPA6010	175.5		0.42	2590	EN14602	136.2		-2.70
2229	CPSC-CH-E1003-09.1	154.775		-1.23	2591	in house	110.82	R(0.01)	-4.72
2232	CPSC-CH-E1003-09.1	172.7		0.20	2597	GB/T 22788	142.1		-2.23
2234	CPSC-CH-E1003-09.1	176.8		0.53	2613	CPSC-CH-E1003-09.1	200.2		2.39
2236	CPSC-CH-E1003-09.1	173.5		0.26	2614	CPSC-CH-E1003-09.1	180.26		0.80
2238	CPSC-CH-E1003-09.1	166		-0.33	2625	CPSC-CH-E1003-09.1	213.45		3.44
2245	CPSC-CH-E1003-09	175.6		0.43	3100	CPSC-CH-E1003-09.1	170.72		0.04
2246	CPSC-CH-E1003-09	185		1.18	3107	CPSC-CH-E1003-09	157.7		-0.99
2247	CPSC-CH-E1003-09.1	154.44		-1.25	3110	CPSC-CH-E1003-09.1	172.40		0.18
2254	CPSC-CH-E1003-09.1	218.0	R(0.05)	3.81	3116	CPSC-CH-E1003-09.1	174.8		0.37
2255	CPSC-CH-E1003-09.1	172.3		0.17	3118	CPSC-CH-E1003-09	155.9		-1.14
2256	CPSC-CH-E1003-09.1	178.15		0.63	3122	CPSC-CH-E1003-09.1	165		-0.41
2258	CPSC-CH-E1003-09	108.61	C,R(0.01)	-4.90	3124	EPA3052	183		1.02
2268	CPSC-CH-E1003-09.1	169.7		-0.04	3146	CPSC-CH-E1003-09.1	186		1.26
2269	in house	174.7		0.36	3151	in house	169.18		-0.08
2277	in house	188.6		1.47	3160	CPSC-CH-E1003-09.1	165.51		-0.37
2286	CPSC-CH-E1003-09.1	165.1		-0.40	3163	in house	190		1.58
2287	ASTM E1645	166.9		-0.26	3167	CPSC-CH-E1003-09.3	165.6		-0.36
2289	CPSC-CH-E1003-09.1	172.3		0.17	3172	CPSC-CH-E1003-09	150.3		-1.58
2290	CPSC-CH-E1003-09.1	169.3		-0.07	3176	CPSC-CH-E1003-09.1	163.26		-0.55
2293	CPSC-CH-E1003-09.1	177.00		0.54	3180	EN1122	147.05		-1.84
2295	CPSC-CH-E1003-09.1	169.95		-0.02	3182	CPSC-CH-E1003-09.1	157.02		-1.05
2296	in house	173.336		0.25	3185	CPSC-CH-E1003-09.1	172.1		0.15
2301	CPSC-CH-E1003-09	164.17		-0.48	3190	CPSC-CH-E1003	162		-0.65
2366	CPSC-CH-E1003-09.1	175.4		0.42	3197	CPSC-CH-E1003-09.1	169.12		-0.08
2367	CPSC-CH-E1003-09.1	176.1		0.47	3199	in house	201.9		2.52
2370	CPSC-CH-E1003-09.1	168		-0.17	3210	CPSC-CH-E1003-09	156		-1.13
2372	CPSC-CH-E1003-09	161.42		-0.70	3214	CPSC-CH-E1003-09.1	177.3		0.57
2375	CPSC-CH-E1003-09.1	178		0.62	3218	CPSC-CH-E1003-09	168.7		-0.12
2380	CPSC-CH-E1003-09.1	174.67		0.36	3220	16CFR1303	127.5	C	-3.40
2385	in house	174		0.30	3228	CPSC-CH-E1003-09.1	172.4		0.18
2390	CPSC-CH-E1003-09.1	149.63		-1.63	3237	CPSC-CH-E1003-09.1	169.62		-0.04
2391	CPSC-CH-E1003-09.1	159.81		-0.82	3242	CPSC-CH-E1003-09.1	185.2		1.20
2409	ASTM F963	173.75		0.28	3248	CPSC-CH-E1003-09.1	180		0.78
2410	CPSC-CH-E1003-09.1	167.71		-0.20	8005	in house	181.8		0.92
2413	CPSC-CH-E1003-09.1	136.1		-2.71	8010	CPSC-CH-E1003-09	183.7048		1.08
2426	CPSC-CH-E1003-09.1	166.6		-0.28					
2429	CPSC-CH-E1003-09	171.0		0.07					
2431	in house	174.2128		0.32					
2433	ASTM E1645	172.69		0.20					
2441	CPSC-CH-E1003-09	175.9		0.46					
2450	CPSC-CH-E1003-09.1	168.0		-0.17					
2452	CPSC-CH-E1001-08.1	96.72	R(0.01)	-5.85					
2453	CPSC-CH-E1003-09.1	177.6		0.59					
2459	CPSC-CH-E1003-09.1	178.22		0.64					
2460		-----		-----					

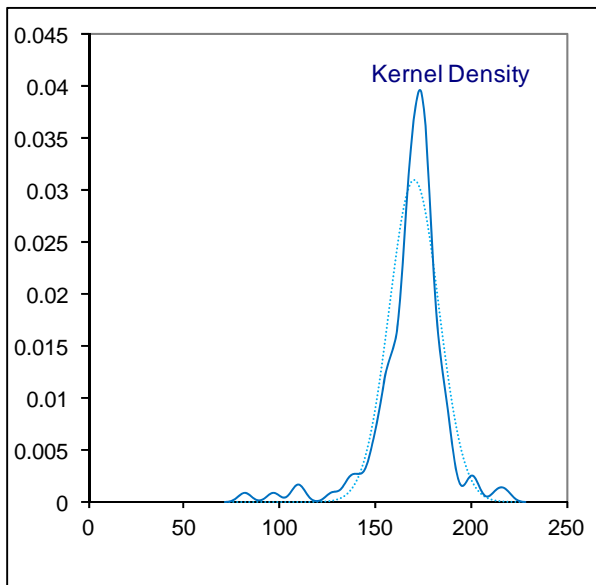
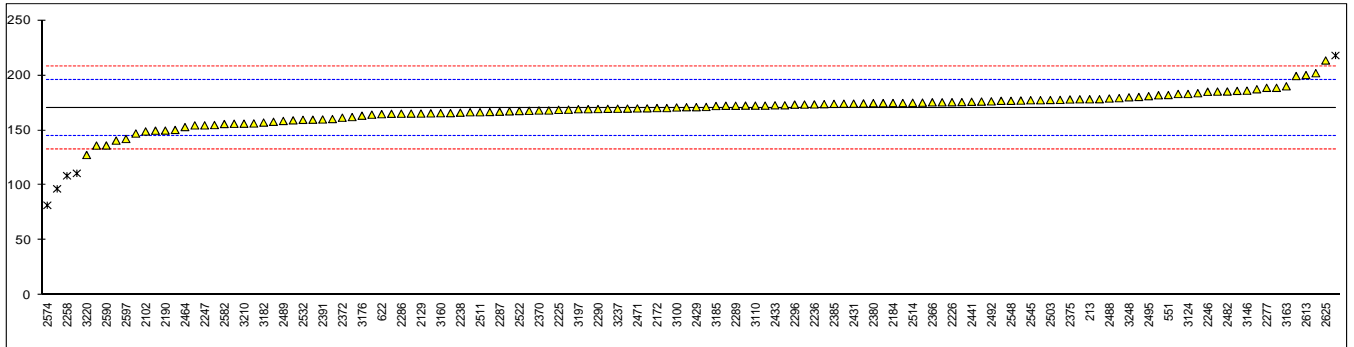
normality
 n
 outliers
 mean (n)
 st.dev. (n)
 R(calc.)
 R(Horwitz)

suspect
 127
 5
 170.177
 12.8905
 36.093
 35.188

Only CPSC-CH-E1003 data:

not OK
 89
 3
 170.328
 11.2590
 31.525
 35.215

Lab 213: first reported 211.043
 Lab 551: first reported 227.55
 Lab 2258: first reported 275.224
 Lab 3220: first reported 238.0



APPENDIX 2

Number of participants per country

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

APPENDIX 3

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
R(0.01)	= outlier in Rosner' outlier test
R(0.05)	= straggler in Rosner' 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, April 2014
- 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/>)
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)