Results of Proficiency Test
Migration of elements
March 2010

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Toy safety is the practice of ensuring that toys, especially those made for children, are safe, usually through the application of set safety standards. In many countries, toys must be able to pass safety tests in order to be sold. Many regions model their safety standards on the EU's EN71 standard, either directly, or through adoption of the ISO 8124 standard which itself is modelled on EN71. In Europe, toys must meet the criteria set by the EC Toy Safety Directive (Council Directive 88/378/EEC). This directive has recently been super-seded by Council Directive 2009/48/EC, which will apply to toy imports into the EU as of 20th July 2011. Part 3 of EN71 describes the determination of migration of elements (metals that are considered hazardous) when a toys gets into contact with an acid solution (0.07 n HCl, simulating gastric acid solution)

In this international interlaboratory study 64 laboratories in 21 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 (PT). Sample preparation and analyses were subcontracted to an ISO17025 accredited laboratory. In this PT 2 different samples of paint with different concentrations of lead, barium and chromium applied on glass plates were used. Participants were requested to report both results of migration before (unrounded) and after analytical correction (ac) *cfr* EN71-3. The 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 two samples used in this proficiency test were prepared by a subcontracted laboratory by the addition of metal salts to a regular paint purchased in Europe. After thorough mixing, the paint was applied to 150 glass plates per sample. The two batches of samples were tested for homogeneity (not migration, but total metal content) on 8 randomly selected samples per batch. The analytical testing was subcontracted to an ISO17025 accredited laboratory. See the following tables for the homogeneity test results.

	Total lead in mg/kg	*Total barium in mg/kg*
Sample #1048-1	192.1	136.6
Sample #1048-2	192.9	139.6
Sample #1048-3	199.1	142.8
Sample #1048-4	186.7	134.4
Sample #1048-5	192.6	138.8
Sample #1048-6	198.1	141.9
Sample #1048-7	194.0	138.5

table 1: measured total lead and total barium for homogeneity test of subsamples #1048 (one outlier excluded)

	Total lead in mg/kg	*Total chromium in mg/kg*
Sample #1049-1	85.5	122.8
Sample #1049-2	89.6	129.8
Sample #1049-3	88.1	128.9
Sample #1049-4	85.6	124.3
Sample #1049-5	88.2	127.4
Sample #1049-6	86.5	126.1
Sample #1049-7	87.3	126.2

table 2: measured total lead and total chromium for homogeneity test of subsamples #1049 (one outlier excluded)

From the test results of tables 1 and 2, the repeatabilities were calculated per metal and subsequently compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO13528, Annex B2 in the next tables:

	Total lead in mg/kg	*Total barium in mg/kg*
r (observed)	11.5	8.1
Reference method	Horwitz	Horwitz
0.3 * R (ref. method)	11.8	8.9

table 3: evaluation of the observed repeatabilities of subsamples #1048

	Total lead in mg/kg	*Total chromium in mg/kg*
r (observed)	4.2	6.9
Reference method	Horwitz	Horwitz
0.3 * R (ref. method)	6.0	8.2

table 4: evaluation of the observed repeatabilities of subsamples #1049

Each calculated repeatability is less than 0.3 times the corresponding reproducibility estimated from the Horwitz equation.

Therefore, homogeneity of the subsamples of #1048 and #1049 was assumed.

Two glass plates (one of each sample #1048 and #1049) were sent to the participating laboratories on February 18, 2010.

2.5 ANALYSES

The participants were requested to determine the migration of elements in accordance with EN71-3, 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.
Both results of migration before and after analytical correction were requested to report.
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 require-ments. Therefore, the z-scores were calculated using a target standard deviation. Due to the lack of precision data in test method EN71-3, the target standard deviation was estimated to be 50% of the analytical correction as specified in paragraph 4.2 of EN71-3. This is justified by the fact that the analytical corrections are based on the uncertainty of the test method and 95% of all results should be within this uncertainty, see appendix D of EN71-3.

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, some problems were encountered. In spite of the extra packaging measures to avoid breakage of the glass plates, five laboratories reported that one or both plates arrived broken. In two cases a plate was broken into two and therefore still could be used. In the other cases a plate was broken into many pieces and consequently new plates were sent to these laboratories.

Only one laboratory decided not to report any results. All other laboratories reported results before the final reporting date.
Finally, the 63 reporting laboratories did report in total 246 numerical results before analytical correction. Observed were 12 statistical outlying results, which is 4.7% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For the migration of each metal a Gaussian distribution was found, except for the migration of lead in sample #1048, in which case the results of the statistical evaluation should be used with care.

4.1 EVALUATION PER SAMPLE AND PER METAL

In this section, the determination is discussed. All statistical results reported on the samples are summarised in appendix 1. The test results before analytical correction were used for the evaluation as not all laboratories applied the analytical correction in the same way, see the tables in appendix 1 and the discussion in chapter 5.

All four reported results of laboratory 2102 were excluded from the statistical calculations because two results proved to be a statistical outlier and the other two results, although no statistical outliers, were suspect, being more or less deviating from the results as reported by the other participants.

Ba on #1048: The migration of barium on this sample, at a very low migration level of 38 mg/kg before analytical correction, may be problematic. However, as the regulatory limit is 1000 mg/kg, this may not be a problem in practice. Three statistical outliers were observed and one false negative result was reported. The observed reproducibility is, after rejection of the statistical outliers, larger than the target reproducibility estimated from the analytical correction.

Pb on #1048: The migration of lead on this sample, at a migration level of 120 mg/kg before analytical correction, is not problematic. Two statistical outliers were observed. The observed reproducibility is, after rejection of the statistical outliers, in good agreement with the target reproducibility estimated from the analytical correction.

Cr on #1049: The migration of chromium on this sample, at a low migration level of 78 mg/kg before analytical correction, is not problematic in general, but it may be problematic for a number of laboratories. Five statistical outliers were observed. The observed reproducibility is, after rejection of the statistical outliers, in good agreement with the target reproducibility estimated from the analytical correction.

Pb on #1049: The migration of lead on this sample, at a low migration level of 52 mg/kg before analytical correction, is not problematic. Two statistical outliers were observed. The observed reproducibility is, after rejection of the statistical outliers, in good agreement with the target reproducibility estimated from the analytical correction.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the target reproducibilities estimated from the analytical correction 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 (50% of the analytical correction*2.8) are compared in the next table.

Parameter	*unit*	*n*	*average*	*2.8 * sd*	*R (target)*
Barium #1048	mg/kg	57	37.95	21.33	15.94
Lead #1048	mg/kg	60	119.27	44.37	50.09
Chromium #1049	mg/kg	56	77.96	14.06	32.74
Lead #1049	mg/kg	59	52.14	17.62	21.90

table 5: reproducibilities of results before analytical correction in samples #1048 and #1049

From the above table it can be concluded that, without statistical calculations, the group of participating laboratories has no difficulties with the determination of the migration of elements in accordance with EN 71-3, when compared with the target reproducibilities estimated from the analytical correction. See also the discussions in paragraphs 4.1.

5 DISCUSSION

When the results of this interlaboratory study are compared to the requirements for toys in EU (table 6), it is noticed that some participants would make different decisions then the majority of the group about the acceptability of the paint for the determined parameters. Twelve laboratories would reject sample #1048 for lead (>90 mg/kg after analytical correction, or 128.57 mg/kg before ac), while 51 laboratories would accept this sample! Sample #1049 would be rejected for chromium by only five laboratories of the group.

	Ba	Cr	Pb
Maximum migrated element in mg/kg toy material	1000	60	90

table 6: maximum migration requirements for toys in EU

It is remarkable to notice that a number of laboratories (11 for barium, 1 for lead in #1048, 1 for chromium and 10 for lead in #1049) did apply the analytical correction only when the test result before analytical correction was above the limit of table 6. The large majority of the laboratories did apply the analytical correction for each test result. This difference in application of the analytical correction explains for the observed large spreads of the results after analytical correcton for barium in sample #1013 and for lead in sample #1014.

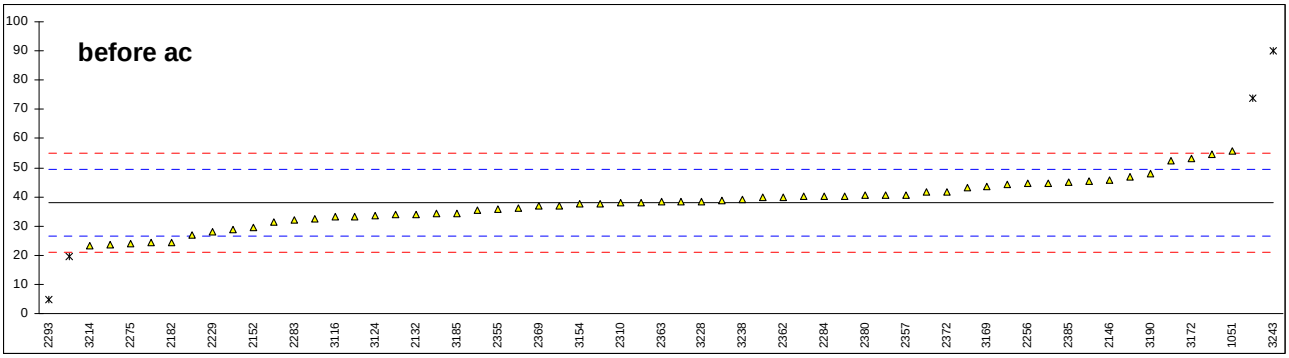
General

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 raise of the quality of the analytical results.

APPENDIX 1

Determination of migration of Barium on sample #1048; results in mg/kg

lab	method	before ac	mark	z(targ)	after ac	mark	ac	remarks
310	EN71-3	31.25	C	-1.18	22	C	30	fr. 62.5 / 44
330	EN71-3	34.3		-0.64	24		30	
622	EN71-3	46.9		1.57	33		30	
1051	EN71-3	55.55		3.09	38.9		30	
2102	EN71-3	19.56	ex	-3.23	13.7	ex	30	see para 4.1
2129	EN71-3	37		-0.17	37		**0**	
2132	EN71-3	34.00		-0.69	23.8		30	
2146	EN71-3	45.6		1.34	32		30	
2152	EN71-3	29.6		-1.47	20.7		30	
2156	EN71-3	52.26		2.51	36.6		30	
2170	EN71-3	44.43		1.14	44.43		**0**	
2172	EN71-3	40.1		0.38	28.1		30	
2182	EN71-3	24.3		-2.40	17.0		30	
2196	EN71-3	26.8		-1.96	19		29	
2201	EN71-3	40.6		0.47	41		**-1**	
2215	EN71-3	41.6		0.64	29.1		30	
2227	EN71-3	23.80		-2.49	16.70		30	
2228	EN71-3	45.500		1.33	31.85		30	
2229	EN71-3	28		-1.75	28		**0**	
2243	EN71-3	37.6		-0.06	26		31	
2245	EN71-3	38.1559		0.04	27		29	
2256	EN71-3	44.73		1.19	-----		**0**	
2275	EN71-3	24.0		-2.45	16.8		30	
2281	EN71-3	43.1816		0.92	30		31	
2283	EN71-3	32	C	-1.04	22	C	31	fr. 30 / 21
2284	EN71-3	40.2		0.40	28		30	
2293	EN71-3	4.6313	G(0.05)	-5.85	3.24	G(0.05)	30	
2294	EN71-3	24.2		-2.42	17		30	
2310	EN71-3	38.04		0.02	26.63		30	
2355	EN71-3	35.9		-0.36	25		30	
2357	EN71-3	40.74		0.49	28.52		30	
2362	EN71-3	40.0		0.36	28.0		30	
2363	EN71-3	38.3		0.06	27		30	
2366	EN71-3	38.40		0.08	26.90		30	
2369	EN71-3	36.8		-0.20	26		29	
2370	EN71-3	40.0		0.36	28.0		30	
2372	EN71-3	41.6		0.64	29.1		30	
2375	EN71-3	35.5		-0.43	24.85		30	
2379	EN71-3	33.85		-0.72	23.7		30	
2380	EN71-3	40.46		0.44	28.3		30	
2385	ICP-OES	45.1		1.26	31.6		30	
3100	EN71-3	28.6		-1.64	20		30	
3110	EN71-3	36.3		-0.29	-----		**0**	
3116	EN71-3	33.03		-0.86	-----		**0**	
3124	EN71-3	33.5		-0.78	-----		---	
3153	EN71-3	32.4		-0.97	-----		---	
3154	DIN-EN71-3	37.47		-0.08	-----		---	
3167	EN71-3	73.8	G(0.01)	6.30	74	G(0.01)	**0**	
3169	EN71-3	43.5856		0.99	-----		---	
3172	EN71-3	53.1		2.66	37.2		30	
3176	EN71-3	44.83		1.21	-----		---	
3179	ICP-OES	33.1		-0.85	23		31	
3185	EN71-3	34.4	C	-0.62	24.1	C	30	fr. Cd 34.4 / 24.1
3190	EN71-3	48		1.77	-----		**0**	
3210		-----		-----	-----		---	
3214	EN71-3	23.34		-2.57	23.3		---	
3216	EN71-3	54.447	C	2.90	-----		---	fr. 60.383
3218	EN71-3	40.2		0.40	40		**0**	
3228	EN71-3	38.4		0.08	26.9		30	
3233	EN71-3	38.9		0.17	39		**0**	
3237		-----		-----	57		---	
3238	ICP-OES	39.0	C	0.19	27.3	C	30	fr. 123.3 / 86.3
3243	ICP-OES	90.2	CG(0.01)	9.18	44.9	G(0.01)	30	fr. 64.2 / 44.9
4095	EN71-3	n.d.		-----	-----		---	false negative?
	normality	OK			not OK			
	n	57			47			
	outliers	3			3			
	mean (n)	37.95			28.20			
	st.dev. (n)	7.616			7.102			
	R(calc.)	21.33			19.89			
	R(target)	15.94			11.81			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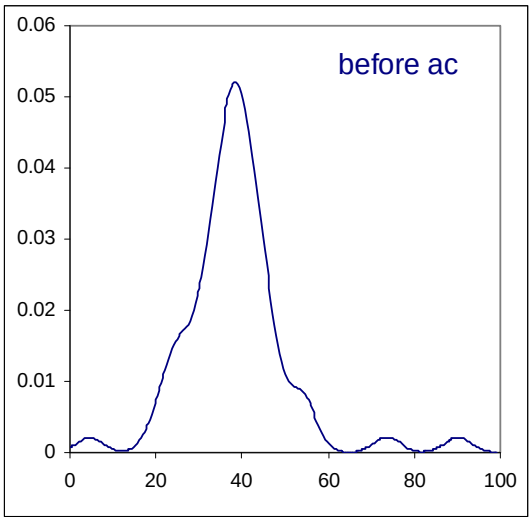
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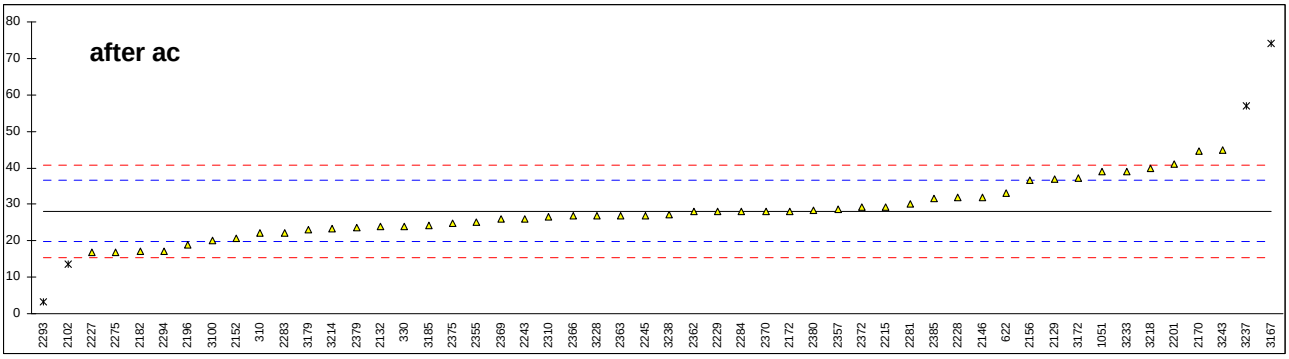
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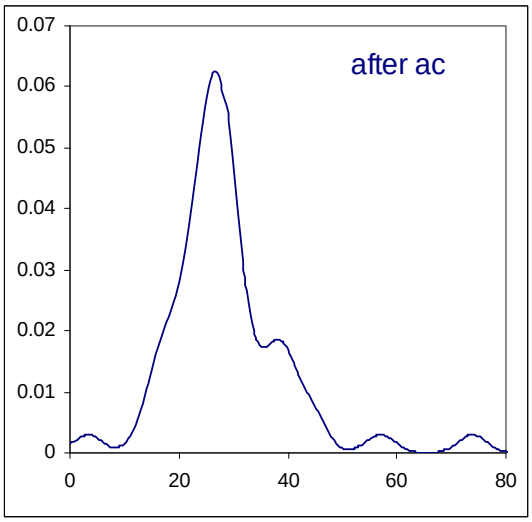
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0.06
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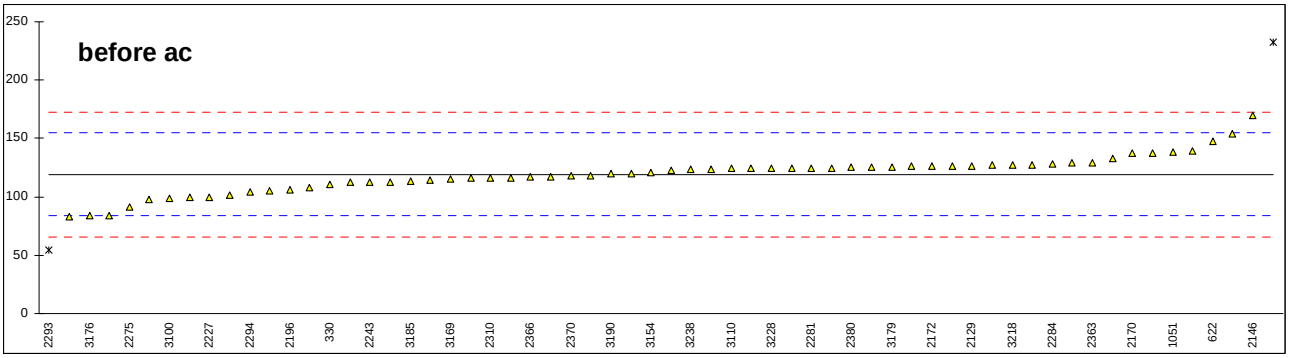
0.03
0.02
0.01
0
0 20 40 60 80

Determination of migration of Lead on sample #1048; results in mg/kg

lab	method	before ac	mark	z(targ)	after ac	mark	ac	remarks
310	EN71-3	101.5	C	-0.99	71	C	30	fr. 203 / 142
330	EN71-3	110.4		-0.50	77		30	
622	EN71-3	147.3		1.57	103		30	
1051	EN71-3	137.99		1.05	96.6		30	
2102	EN71-3	232.74	G(0.01)	6.34	162.9	G(0.01)	30	
2129	EN71-3	126.2		0.39	120		**5**	
2132	EN71-3	99.75		-1.09	69.8		30	
2146	EN71-3	169.7		2.82	119		30	
2152	EN71-3	114.1		-0.29	79.9		30	
2156	EN71-3	127.6		0.47	89.3		30	
2170	EN71-3	137.21		1.00	96.05		30	
2172	EN71-3	126.2		0.39	88.3		30	
2182	EN71-3	82.6		-2.05	57.8		30	
2196	EN71-3	105.7		-0.76	74		30	
2201	EN71-3	125.0		0.32	87.5		30	
2215	EN71-3	126		0.38	88.2		30	
2227	EN71-3	99.85		-1.09	69.9		30	
2228	EN71-3	154.250		1.96	107.98		30	
2229	EN71-3	108		-0.63	75.6		30	
2243	EN71-3	112.6		-0.37	79		30	
2245	EN71-3	118.424		-0.05	83		30	
2256	EN71-3	115.98		-0.18	81.19		30	
2275	EN71-3	91.5		-1.55	64.0		30	
2281	EN71-3	124.844		0.31	87		30	
2283	EN71-3	98	C	-1.19	69	C	30	fr. 95 / 67
2284	EN71-3	128.4		0.51	90		30	
2293	EN71-3	54.2633	G(0.01)	-3.63	37.98	G(0.01)	30	
2294	EN71-3	103.9		-0.86	73		30	
2310	EN71-3	116		-0.18	81.2		30	
2355	EN71-3	123.9		0.26	87		30	
2357	EN71-3	127.65		0.47	89.34		30	
2362	EN71-3	137.7		1.03	96.4		30	
2363	EN71-3	129.6		0.58	91		30	
2366	EN71-3	116.73		-0.14	81.7		30	
2369	EN71-3	126.2		0.39	88		30	
2370	EN71-3	118		-0.07	82.6		30	
2372	EN71-3	122.3		0.17	85.6		30	
2375	EN71-3	112.6		-0.37	78.82		30	
2379	EN71-3	120.05		0.04	84.0		30	
2380	EN71-3	125.143		0.33	87.6		30	
2385	ICP-OES	124.8		0.31	87.4		30	
3100	EN71-3	98.6		-1.16	69		30	
3110	EN71-3	124.4		0.29	87.1		30	
3116	EN71-3	116.20		-0.17	81.34		30	
3124	EN71-3	117		-0.13	-----		---	
3153	EN71-3	105.3		-0.78	-----		---	
3154	DIN-EN71-3	121.00		0.10	-----		---	
3167	EN71-3	125.6		0.35	88		30	
3169	EN71-3	114.9013		-0.24	-----		---	
3172	EN71-3	124.5		0.29	87.2		30	
3176	EN71-3	83.55		-2.00	-----		---	
3179	ICP-OES	125.7		0.36	88		30	
3185	EN71-3	113.9		-0.30	79.7		30	
3190	EN71-3	120		0.04	84		30	
3210		-----		-----	-----		---	
3214	EN71-3	84.07		-1.97	84.1		---	
3216	EN71-3	138.889		1.10	-----		---	
3218	EN71-3	127.6		0.47	89		30	
3228	EN71-3	124.5		0.29	87.2		30	
3233	EN71-3	132.5		0.74	93		30	
3237		-----		-----	100.8		---	
3238	ICP-OES	123.3	C	0.23	86.3	C	30	fr. 39.0 / 27.3
3243	ICP-OES	129		0.54	90.3		30	
4095	EN71-3	112.2		-0.40	78.5		30	
	normality	not OK			not OK			
	n	60			55			
	outliers	2			2			
	mean (n)	119.27			85.30			
	st.dev. (n)	15.846			11.518			
	R(calc.)	44.37			32.25			
	R(target)	50.09			35.82			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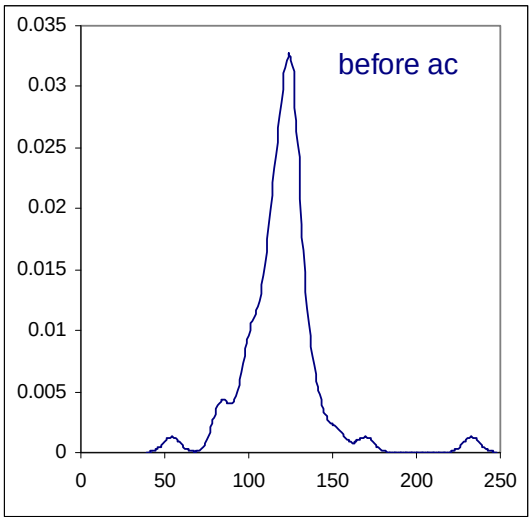
250
200
150
100
50
0
2293 3176 2275 3100 2227 2294 2196 330 2243 3185 3169 2310 2366 2370 3190 3154 3238 3110 3228 2281 2380 3179 2172 2129 3218 2284 2363 2170 1051 622 2146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0.03
0.025
0.02
0.015
0.01
0.005
0
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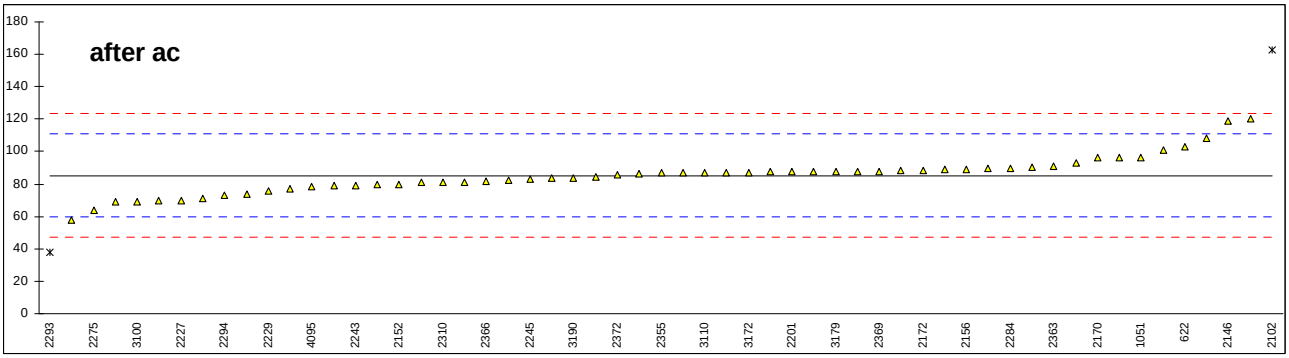
after ac
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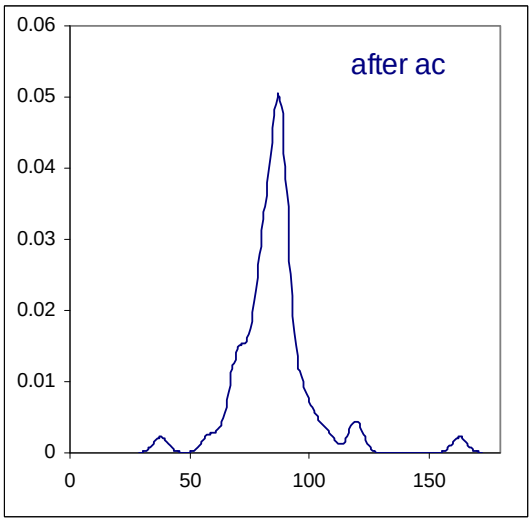
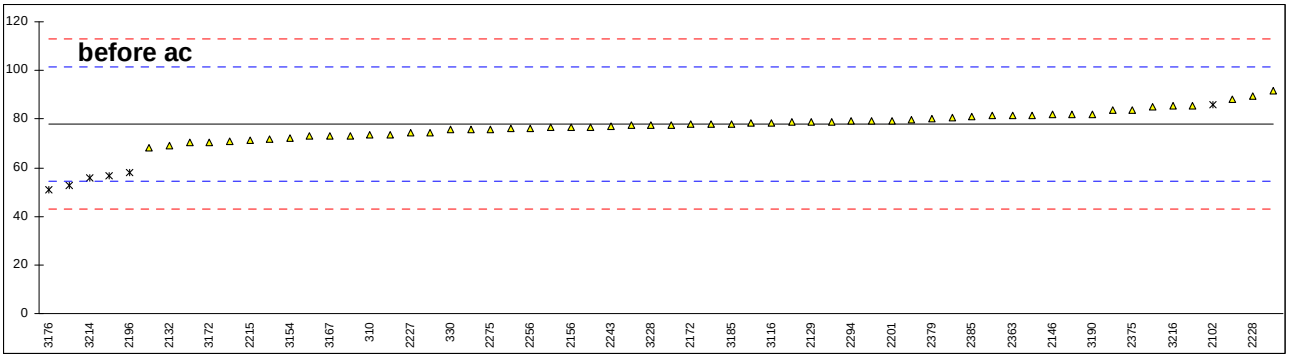
160
140
120
100
80
60
40
20
0
2293 2275 3100 2227 2294 2229 4095 2243 2152 2310 2366 2245 3190 2372 2355 3110 3172 2201 3179 2369 2172 2156 2284 2363 2170 1051 622 2146 2102


after ac
0.06
0.05
0.04
0.03
0.02
0.01
0
0 50 100 150

Determination of migration of Chromium on sample #1049; results in mg/kg

lab	method	before ac	mark	z(targ)	after ac	mark	ac	remarks
310	EN71-3	73.5	C	-0.38	51.5	C	30	fr. 147 / 103
330	EN71-3	75.5		-0.21	53		30	
622	EN71-3	52.5	DG(0.01)	-2.18	37	G(0.05)	30	
1051	EN71-3	83.62		0.48	58.5		30	
2102	EN71-3	85.73	ex	0.66	60	ex	30	see para 4.1
2129	EN71-3	79		0.09	80	G(0.01)	**-1**	
2132	EN71-3	69.25		-0.74	48.5		30	
2146	EN71-3	81.7		0.32	57		30	
2152	EN71-3	73.7		-0.36	51.6		30	
2156	EN71-3	76.42		-0.13	53.5		30	
2170	EN71-3	87.94		0.85	61.56		30	
2172	EN71-3	77.8		-0.01	54.5		30	
2182	EN71-3	68.0		-0.85	47.6		30	
2196	EN71-3	58.1	G(0.05)	-1.70	41	G(0.05)	29	
2201	EN71-3	79.4		0.12	55.6		30	
2215	EN71-3	71.4		-0.56	50.0		30	
2227	EN71-3	74.30		-0.31	52.00		30	
2228	EN71-3	89.600		1.00	62.72		30	
2229	EN71-3	85		0.60	59.5		30	
2243	EN71-3	77.2		-0.06	54		30	
2245	EN71-3	77.4899		-0.04	54		30	
2256	EN71-3	76.03		-0.16	53.22		30	
2275	EN71-3	75.7		-0.19	53.0		30	
2281	EN71-3	78.9454		0.08	55		30	
2283	EN71-3	71	C	-0.60	50	C	30	fr. 66 / 46
2284	EN71-3	91.7		1.18	64		30	
2293	EN71-3	56.6833	DG(0.01)	-1.82	39.68	G(0.05)	30	
2294	EN71-3	79.1		0.10	55		30	
2310	EN71-3	74.32		-0.31	52.03		30	
2355	EN71-3	76.0		-0.17	53		30	
2357	EN71-3	80.70		0.23	56.49		30	
2362	EN71-3	81.4		0.29	57.0		30	
2363	EN71-3	81.3		0.29	57		30	
2366	EN71-3	76.58		-0.12	53.6		30	
2369	EN71-3	79.6		0.14	56		30	
2370	EN71-3	75.6		-0.20	52.9		30	
2372	EN71-3	70.5		-0.64	49.4		30	
2375	EN71-3	83.9		0.51	58.73		30	
2379	EN71-3	79.98		0.17	56.0		30	
2380	EN71-3	73.219		-0.41	51.3		30	
2385	ICP-OES	80.9		0.25	56.6		30	
3100	EN71-3	79.0		0.09	55		30	
3110	EN71-3	85.6		0.65	59.9		30	
3116	EN71-3	78.55		0.05	54.99		30	
3124	EN71-3	76.4		-0.13	-----		---	
3153	EN71-3	73.0		-0.42	-----		---	
3154	DIN-EN71-3	72.045		-0.51	-----		---	
3167	EN71-3	73.2		-0.41	51		30	
3169	EN71-3	78.1831		0.02	-----		---	
3172	EN71-3	70.5		-0.64	49.4		30	
3176	EN71-3	50.87	DG(0.01)	-2.32	-----		---	
3179	ICP-OES	81.3		0.29	57		30	
3185	EN71-3	77.9		0.00	54.5		30	
3190	EN71-3	82		0.35	57		30	
3210		-----		-----	-----		---	
3214	EN71-3	55.62	DG(0.01)	-1.91	55.6		---	
3216	EN71-3	85.5715		0.65	-----		---	
3218	EN71-3	77.3		-0.06	54		30	
3228	EN71-3	77.4		-0.05	54.2		30	
3233	EN71-3	77.9		0.00	55		29	
3237		-----		-----	70.1	G(0.05)	---	
3238	ICP-OES	81.8	C	0.33	57.2	C	30	fr. Cd 81.8 / 57.2
3243	ICP-OES	71.6		-0.54	50.1		30	
4095	EN71-3	79.1		0.10	55.4		30	
	normality	OK			OK			
	n	56			51			
	outliers	5			5			
	mean (n)	77.96			54.62			
	st.dev. (n)	5.023			3.499			
	R(calc.)	14.06			9.80			
	R(target)	32.74			22.94			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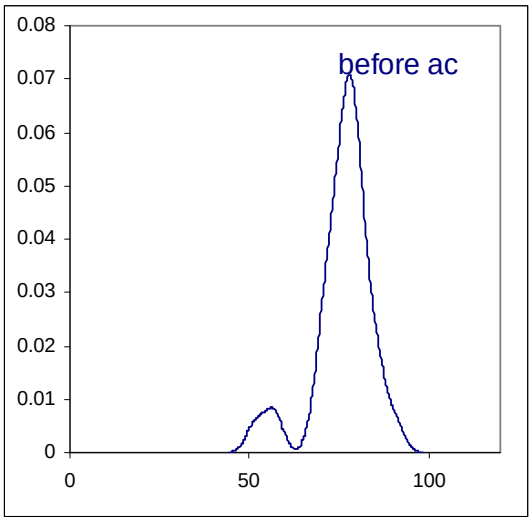
80
60
40
20
0
3176
3214
2196
2132
3172
2215
3154
3167
310
2227
330
2275
2256
2156
2243
3228
2172
3185
3116
2129
2294
2201
2379
2385
2363
2146
3190
2375
3216
2102
2228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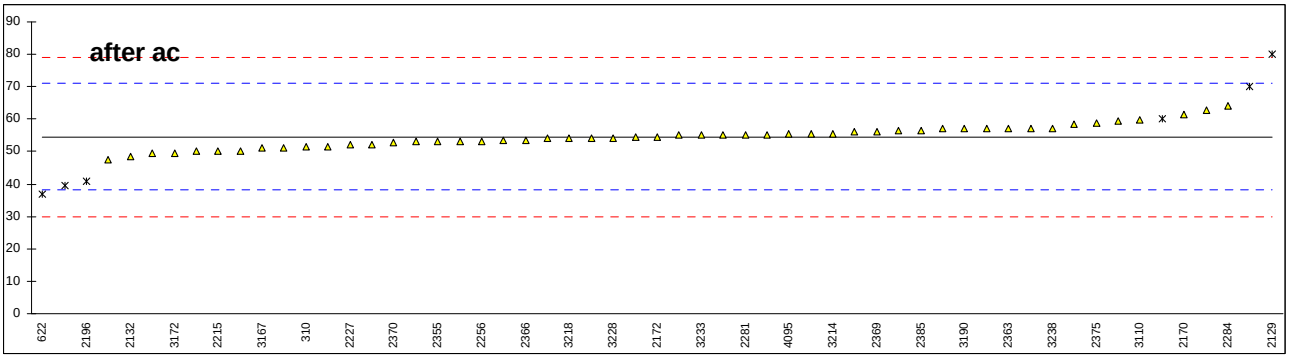
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
0
50
100


after ac
90
80
70
60
50
40
30
20
10
0
622
2196
2132
3172
2215
3167
310
2227
2370
2355
2256
2366
3218
3228
2172
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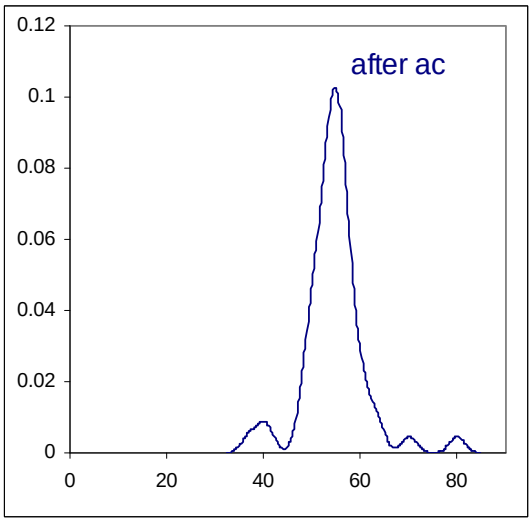
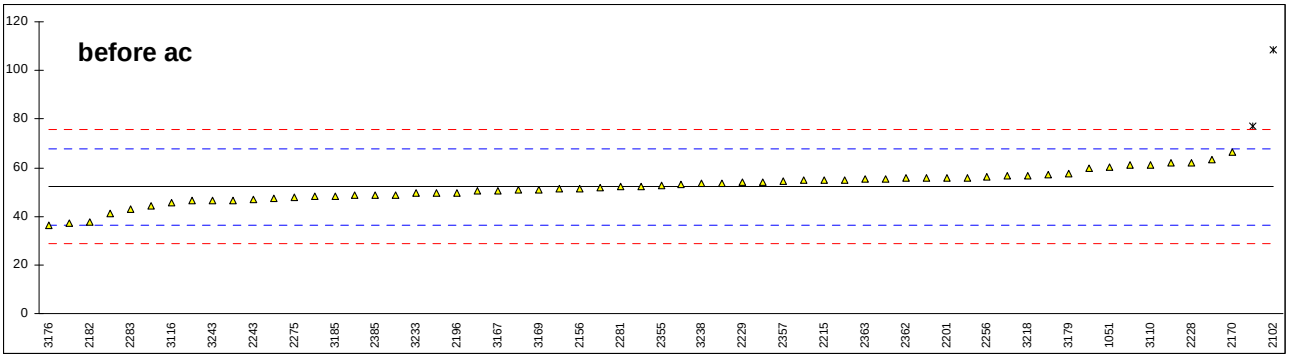
2363
3238
2375
3110
2170
2284
2129


after ac
0.12
0.1
0.08
0.06
0.04
0.02
0
0
20
40
60
80

Determination of migration of Lead on sample #1049; results in mg/kg

lab	method	before ac	mark	z(targ)	after ac	mark	ac	remarks
310	EN71-3	47.5	C	-0.59	33.5	C	29	fr. 95 / 67
330	EN71-3	51.7		-0.06	36		30	
622	EN71-3	63.4		1.44	44		31	
1051	EN71-3	60.44		1.06	42.3		30	
2102	EN71-3	108.45	G(0.01)	7.20	75.9	G(0.01)	30	
2129	EN71-3	61		1.13	60	DG(0.05)	**2**	
2132	EN71-3	44.25		-1.01	31.0		30	
2146	EN71-3	55.5		0.43	39		30	
2152	EN71-3	41.0		-1.42	28.7		30	
2156	EN71-3	51.4		-0.09	36		30	
2170	EN71-3	66.47		1.83	66.47	G(0.05)	**0**	
2172	EN71-3	54.8		0.34	38.4		30	
2182	EN71-3	37.7		-1.85	26.4		30	
2196	EN71-3	49.8		-0.30	35		30	
2201	EN71-3	55.9		0.48	55.9		**0**	
2215	EN71-3	55.0		0.37	38.5		30	
2227	EN71-3	46.35		-0.74	32.4		30	
2228	EN71-3	62.200		1.29	43.54		30	
2229	EN71-3	54		0.24	54		**0**	
2243	EN71-3	46.9		-0.67	33		30	
2245	EN71-3	51.2864		-0.11	36		30	
2256	EN71-3	56.08		0.50	-----		**0**	
2275	EN71-3	48.0		-0.53	33.6		30	
2281	EN71-3	52.0813		-0.01	36		31	
2283	EN71-3	43	C	-1.17	30	C	30	fr. 32 / 22
2284	EN71-3	61.9		1.25	43		31	
2293	EN71-3	<5		< 6.03	<5		---	false negative?
2294	EN71-3	53.2		0.14	37		30	
2310	EN71-3	49.66		-0.32	34.77		30	
2355	EN71-3	52.7		0.07	37		30	
2357	EN71-3	54.50		0.30	38.15		30	
2362	EN71-3	55.6		0.44	38.9		30	
2363	EN71-3	55.5		0.43	39		30	
2366	EN71-3	48.14		-0.51	33.7		30	
2369	EN71-3	55.0		0.37	39		29	
2370	EN71-3	51.0		-0.15	35.7		30	
2372	EN71-3	50.3		-0.24	35.2		30	
2375	EN71-3	54.2		0.26	37.94		30	
2379	EN71-3	52.10		-0.01	36.5		30	
2380	EN71-3	55.842		0.47	39.1		30	
2385	ICP-OES	48.8		-0.43	34.2		30	
3100	EN71-3	48.6		-0.45	34		30	
3110	EN71-3	61.3		1.17	-----		**0**	
3116	EN71-3	45.61		-0.83	-----		**0**	
3124	EN71-3	53.7		0.20	-----		---	
3153	EN71-3	48.9		-0.41	-----		---	
3154	DIN-EN71-3	46.675		-0.70	-----		---	
3167	EN71-3	50.7		-0.18	36		29	
3169	EN71-3	51.0526		-0.14	-----		---	
3172	EN71-3	56.6		0.57	39.6		30	
3176	EN71-3	36.53		-2.00	-----		---	
3179	ICP-OES	57.7		0.71	40		31	
3185	EN71-3	48.4		-0.48	33.9		30	
3190	EN71-3	56		0.49	-----		**0**	
3210		-----		-----	-----		---	
3214	EN71-3	37.10		-1.92	37.1		---	
3216	EN71-3	59.901		0.99	-----		---	
3218	EN71-3	56.6		0.57	57	DG(0.05)	**-1**	
3228	EN71-3	57.2		0.65	40.0		30	
3233	EN71-3	49.6		-0.32	50		**-1**	
3237		-----		-----	48.5		---	
3238	ICP-OES	53.5		0.17	37.5		30	
3243	ICP-OES	46.4		-0.73	32.5		30	
4095	EN71-3	77.0	G(0.05)	3.18	53.9		30	
	normality	OK			not OK			
	n	59			48			
	outliers	2			4			
	mean (n)	52.14			38.03			
	st.dev. (n)	6.293			6.177			
	R(calc.)	17.62			17.30			
	R(target)	21.90			15.97			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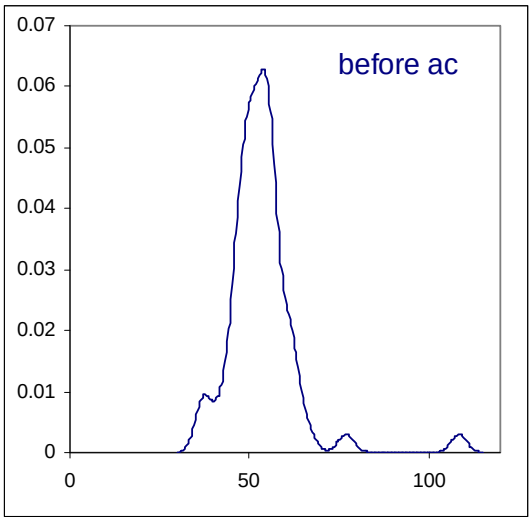
120
100
80
60
40
20
0
3176 2182 2283 3116 3243 2243 2275 3185 2385 3233 2196 3167 3169 2156 2281 2355 3238 2229 2357 2215 2363 2362 2201 2256 3218 3179 1051 3110 2228 2170 2102


before ac
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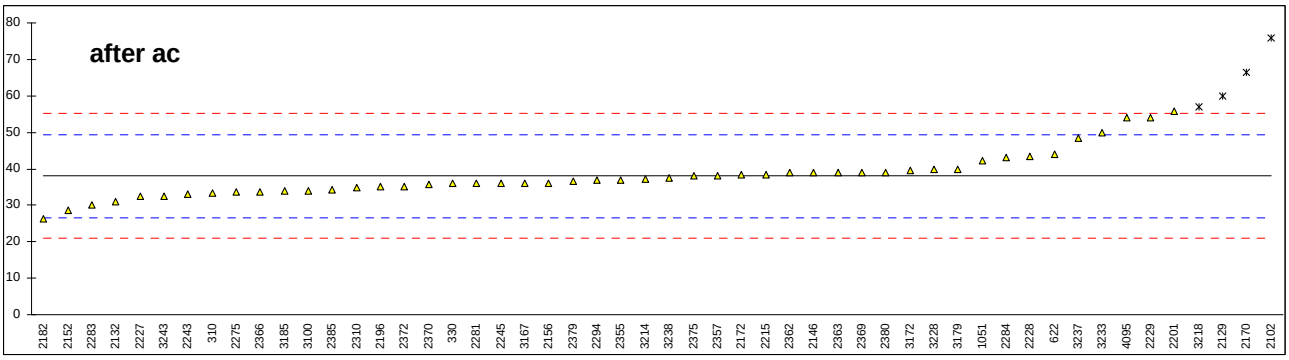
0.06
0.05
0.04
0.03
0.02
0.01
0
0
50
100


after ac
80
70
60
50
40
30
20
10
0
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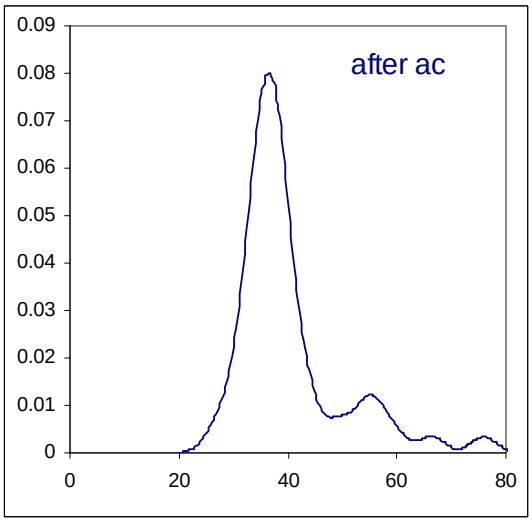
after ac
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
0
20
40
60
80

APPENDIX 2

Number of participants in alphabetic country order:

1 lab in	BANGLADESH
1 lab in	DENMARK
1 lab in	FINLAND
4 labs in	FRANCE
5 labs in	GERMANY
1 lab in	GUATEMALA
9 labs in	HONG KONG
1 lab in	HUNGARY
1 lab in	INDIA
1 lab in	INDONESIA
1 lab in	ITALY
1 lab in	MALAYSIA
2 labs in	MEXICO
23 labs in	P.R. of CHINA
1 lab in	SINGAPORE
1 lab in	SPAIN
3 labs in	TAIWAN R.O.C.
1 lab in	THAILAND
2 labs in	THE NETHERLANDS
3 labs in	TURKEY
1 lab in	U.S.A.

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
n.a.	= not applicable
n.d.	= not detected
fr.	= first reported result
ac	= analytical correction *cfr* EN71-3, paragraph 4.2

Literature:

1 i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
2 Council Directive 88/378/EEC
3 Council Directive 2009/48/EC
4 EN71-3 Migration of certain elements
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 ISC7/GF/csteeop/toysinorg/220604 D(04) Assessment of bioavailability of certain elements in toys
11 ISO13528:2005 Statistical methods for use in proficiency testing by interlaboratory comparisons
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/).