



**Asia Pacific Laboratory Accreditation Cooperation
Proficiency Testing Programme**

APLAC T098

Determination of Migration of Heavy Metals in Toys Coating

FINAL REPORT

April 2016



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Summary

1. This proficiency testing programme (APLAC T098) was organized by China National Accreditation Service for Conformity Assessment (CNAS) in collaboration with Guangdong Inspection and Quarantine Technology Center-Toys and Juvenile Products Testing Lab (IQTC), under the auspices of Asia-Pacific Laboratory Accreditation Cooperation (APLAC).
2. A total of 86 laboratories from 39 economies registered for the programme. 81 laboratories submitted their results on time (table 1).
3. Assigned values and standard deviations are determined by the robust analysis according to Algorithm A, in Annex C of ISO 13528. The z-score is used as the numerical indicator to assess individual participant's performance in the programme.
4. Summary of participants' statistical results as followed (unit:mg/kg).

	Migration of Barium	Migration of Cadmium	Migration of Chromium	Migration of Lead
Number of results	71	78	74	79
x*	298	20.4	25.4	74.6
s*	82.6	6.10	6.16	24.0
CV%	27.7	29.9	24.2	32.1
Max	529.6	73.5	106.38	173.46
Min	142.4	6.475	12.3	30.275
Range	387.2	67.025	94.08	143.185

Note: The extreme outliers from lab 008, 058, 072 are not included in the statistical analysis.

5. Summary of the z-scores of the Participants as follow.

z-score	Number of results (Percentage)			
	Migration of Barium	Migration of Cadmium	Migration of Chromium	Migration of Lead
z ≤2.0	67(94%)	69(88%)	67(91%)	73(92%)
2.0 < z <3.0	4(5.6%)	2(2.6%)	6(8.1%)	3(3.8%)
z ≥3.0	0(0.0%)	7(9.0%)	1(1.4%)	3(3.8%)
Total	71(100%)	78(100%)	74(100%)	79(100%)

1. Introduction

1.1 This report summarizes the results of the APLAC T098 proficiency testing programme on the routine analysis of Migration of Heavy Metals in Toys Coating. The programme was organized by China National Accreditation Service for Conformity Assessment (CNAS) in collaboration with Guangdong Inspection and Quarantine Technology Center-Toys and Juvenile Products Testing Lab (IQTC), under the auspices of Asia-Pacific Laboratory Accreditation Cooperation (APLAC). IQTC has been accredited against ISO/IEC 17043 (CNAS PT0022) for the scope of this PT program.

1.2 Barium, Cadmium, Chromium and Lead are toxic metals which can have harmful effects on the behavior and development of children even at very low levels of exposure.

Once heavy metals enter the body, they compete with and displace essential minerals such as zinc, copper, magnesium, and calcium, and interfere with organ system function. Lead and cadmium are human poisons. Chromium is required by the body in small amounts, but can be toxic in larger quantities. Barium sulfate is basically nontoxic, whereas barium salts are rapidly absorbed and cause profound, potentially fatal hypokalemia. Young children are more susceptible to the effects of lead exposure because they absorb several times the percent ingested compared with adults and because their brains are more plastic and even brief exposures may influence developmental processes.

There are many similar toys regulatory such as ISO 8124-3:2010+A1:2014, EN71-3:2013+A1:2014, GB 6675.4-2014, ASTM F963-11 with respect to heavy metal migration in children's toys in different countries and areas. Currently, the standard concerning migration of certain elements in toys used in China is GB 6675.4-2014, it was drafted in accordance with ISO 8124.3:2010. The maximum acceptable migration of certain elements are given below.

Toy material	Element							
	Sb	As	Ba	Cd	Cr	Pb	Hg	Se
Other toy materials, except modelling clay and finger paint	60	25	1000	75	60	90	60	500

This program is intended to understand the general level in determining migration of heavy metals in toys coating, and to find out and identify the diversity among laboratories. It can also help the specific laboratories to make improvement, and try to establish a kind of equivalence.

1.3 Each participant was confidentially assigned with a unique laboratory code (001 to 086) which is used throughout the programme.

1.4 The programme was carried out as the schedule below.

Event	Planned	Responsible
Invitation	January 2015 - April 2015	CNAS
Dispatch of PT samples	May 2015	IQTC
Submit the results	June 2015	CNAS/IQTC
Distribution of Interim report	Sep. 2015	CNAS/IQTC
Draft final report	June-December 2015	CNAS/IQTC

2. Objective

2.1 The main objective of the scheme is to evaluate the performance of participating laboratories on the analysis of Migration elements of Barium, Cadmium, Chromium and Lead in Toys Coating by means of inter-laboratory comparison. In this programme “proficiency” is taken to mean the performance of agreement with other participants.

2.2 Test results from participants were evaluated according to the statistical techniques outlined in ISO 13528:2015 *Statistical methods for use in proficiency testing by inter laboratory comparisons*. The z-score was used as a numerical indicator to assess individual participant’s performance in the scheme.

3. Proficiency Testing Items

3.1 Each registered participant was provided with a brown bottle labeled H1504A. Each bottle contains about 2g of toy painting powder which has already been crushed and passed through 500 µm sieve.

3.2 The homogeneity study and stability study of the test material was carried out by IQTC in accordance with the procedures stipulated in APPENDIX B. The results indicated that the test material was considered to be sufficiently homogeneous and stable for the proficiency testing programme.

3.3 The samples along with relevant documents were distributed by express in May 2015. Relevant documents were also provided via e-mail, which can be referred to APPENDIX C.

3.4 Upon receipt of the proficiency test samples, participants were required to immediately check the physical conditions of the bottles and promptly acknowledge IQTC by returning the “Sample Receipt Form” via the e-mail box of the contact. Replacement would be arranged if the proficiency test sample was identified to be not suitable for analysis.

4. Test Required

Participants were required to:

- a) Report both of the single measurement result and average result of the 4 test items/parameters using their routine test methods; and not versions of the method specially adapted for the proficiency test. It is the responsibility of participating laboratories to avoid collusion or falsification of results;
- b) Report the analytical results to one decimal place in the “Result Proforma” (for example: XXX.X or XX.X);
- c) Provide detailed information of the test methods/conditions applied in this scheme, and
- d) Report the Expanded measurement uncertainty and Method Detection Limit for each result as possible as they can.

5. Statistical Evaluation

5.1 Participants’ performance was evaluated via a numeric indicator, z-score, which is calculated as follows:

$$z = \frac{x - x^*}{s^*}$$

Where x = the reported average result of the participant

x^* = the robust average according to ISO 13528:2015 , Algorithm A, in Annex C

s^* = the robust standard deviation according to ISO 13528:2015, Algorithm A, in Annex C

5.2 The z-score is commonly interpreted as follows:

$ z \leq 2.0$	Satisfactory	
$2.0 < z < 3.0$	Questionable	Warning Signal
$ z \geq 3.0$	Unsatisfactory	Action Signal

5.3 Participants with the action signal should thoroughly investigate their results and find the possible root cause. Participants are encouraged to review their results if with warning signal.

5.4 In addition to tables of the results, z-scores, and summary statistics, the ordered z-score histograms are included in the report. On these charts each laboratory's z-score is shown in order of magnitude, and the lab with $|z| \geq 3.0$ were marked with its lab code on the graph. From these charts each laboratory can readily compare its performance relative to other laboratories.

6. Results Reported by Participants

6.1 Participants' results

6.1.1 An overview of participants' statistical results is summarized in Table 2 and participants with unsatisfactory results are summarized in Table 3.

6.1.2 Test results received from participants are presented in APPENDIX A.

6.1.3 Participants' results of Migration of Barium, Migration of Cadmium are given in Table A.1, results of Migration of Chromium and Migration of Lead are given in Table A.2.

6.2 Statistical analysis of participants' results

6.2.1 The normal distribution check was taken out before statistical analysis. Anderson-Darling method was applied to check the normal distribution of the participants' result. And Grubb's method was applied to check the outlier before Anderson-Darling analysis. Relevant graphs of normality tests are given in Figure 1 to Figure 4. Kernel density plots, presented in Figure 5 to Figure 8, are more visual than normal probability plots by applying maximum smoothing strategy. This is a statistical calculation method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. It further displays the rough normal distributions of the results. The equation $\sigma_k = 0.9s^*/p^{0.2}$ where s^* is a robust standard deviation used according to ISO 13528:2015.

6.2.2 The Scatter Diagram for Migration of Barium, Migration of Cadmium, Migration of Chromium and Migration of Lead with Uncertainty are presented in Figure 9 to Figure 12, in which the relative location of each participant's test results was marked intuitively.

6.2.3 The number and percentage of z-scores in the satisfactory range ($|z| \leq 2.0$), questionable range ($2.0 < |z| < 3.0$) and unsatisfactory range ($|z| \geq 3.0$) are summarized in Table 4.

6.2.4 The z-scores of Participants' results of Migration of Barium, Migration of Cadmium, Migration of Chromium and Migration of Lead are presented graphically in Figure A.1 to Figure A.4. A bar chart of total four migration elements is presented graphically in Figure A.5.

6.2.5 Interim report of APLAC T098 has been distributed to both of the participants and accreditation bodies by email on Sep 18th, 2015, which included the summary of statistical results, individual Z-score and some important testing parameters. The corresponding opinions collected have been closed by Oct 15th, 2015.

6.2.6 Gross stragglers were taken out before statistical analysis. Concentration of Cr reported by Lab 058 is the sum of two replicates, the methods used by Lab 072 and 008 are not suitable, so the result of Cr(lab code 058) and all the elements of lab code 008&072 are removed from the data set before statistical evaluation, and these results are all considered unsatisfactory. As a result, small distinctions have occurred in the final evaluation and individual Z-scores compared to those in the interim report.

6.3 Testing parameters of participants

6.3.1 According to the information from *results proforma*, the testing parameters(e.g. volume, sample weight, acid volume, shaking frequency and testing instrument) of Barium, Cadmium, Chromium and Lead are summarized in Table A.3 of APPENDIX A.

6.3.2 From the information kindly offered by participants, it can be concluded that ISO and EN standards are prevailing methods that participants used. Other methods may be traced back to them (e.g. GB standards involved in this programme are derived from ISO standards). Overview of analysis methods used by participants is summarized in Table 5 and Table A.3 of APPENDIX A.

6.4 Measurement uncertainty

6.4.1 Although the measurement uncertainties reported by participants were not requested to be used in assessing the performance of participants, it was one of the important technical requirements of ISO/IEC 17025:2005. Scatter diagrams with uncertainty were presented in Figure 9 to Figure 12, lab codes of those z-score $\geq$ 3.0 and z-score $\leq$ -3.0 were marked on the graphs below. All uncertainties were plotted in the Figures, except the situation that some participants did not report uncertainties.

6.4.2 Some participants didn't report measurement uncertainties. The number of participants reporting measurement uncertainties is given in Table 7.

6.4.3 Uncertainty of assigned value

Because assigned values which are derived from the participant results are not traceable to an international measurement standard, the uncertainty of assigned value is estimated from the participant results. According to ISO 13528:2015, the standard uncertainty of assigned value is calculated as the following formula:

$$u_x = \frac{1.25 \times s^*}{\sqrt{p}}$$

It shows that the uncertainties of the assigned values are small enough compared with the $0.3s^*$. Details of uncertainty are summarized in Table 8.

7. Comments

7.1 Influence of Standard Methods

7.1.1 The analysis methods used by participants is summarized in Table 5. Different methods and corresponding number of unsatisfactory results are summarized according to items respectively. Detailed information is summarized in Table 6.

7.1.2 The shaking frequency in the migration procedure is not specific requirement in ISO&EN&GB&ASTM standards. The shaking frequency offered by participants is summarized in Table 9.

7.1.3 Some mistakes were found from the information the participants provided. For example, CPSC-CH-E1003-09 was used for digesting sample in Lab 008. But this standard is an operating procedure for determining total lead (Pb) in paint and other similar surface coatings, which is not consistent with ISO 8124-3 standard for this proficiency testing. Also, nitric acid was used to migrate sample in Lab 072, which is also not consistent with the requirements of EN 71-3:2013+A1:2014 that used by the most participants. The result of migration of chromium from Lab 058 is the sum value rather than the average value of the two testing results, which is an obvious writing mistake. The participants are encouraged to investigate the reason(s) the same as the warning signal was given out.

7.1.4 According to the migration procedure of ISO and EN standards, 50 times acid should be added to the test portion. Some participants (Lab 009,021,038,040,046,068,076) did not follow this requirement.

7.2 Analyzing of measurement uncertainty

Further laboratory capacity building efforts in respect of the estimation of measurement uncertainty are recommended for the participants who showed some difficulties in reporting measurement uncertainty (either the measurement uncertainty was not reported or the reported measurement uncertainty was underestimated or overestimated). Following the idea from the latest version of ISO 13528, u_{max} could be $1.5s^*$. u_{min} could vary in some range due to different methods. Those participants with uncertainty larger than $1.5s^*$ are encouraged to follow *GUM (Guide to the Expression of Uncertainty in Measurement)*, in quantifying measurement uncertainty in analytical measurements.

7.3 Influence of measuring apparatus

From the information kindly offered by participants, it can be concluded that ICP-OES, ICP-MS and AAS are prevailing apparatus that participants used. Overview of analysis apparatus used by participants is summarized in Table 10.

7.4 Analyzing of repeatability

Each participant made two replicate determinations under repeatability conditions. A review has indicated that the difference between the replicates has exceeded two times of measurement uncertainty from some laboratories. It's encouraged to investigate the reason(s) by the participants.

7.5 Analyzing of kernel density plots

It is difficult to infer from independent information that why there is a minor mode. In Figure 6 and Figure 7, the minor modes are well separated from the main body of the data, so the minor modes can be attributed to outlying results, and the area of the minor modes is very small compared to the total area. Then robust statistic is still used to assign the value according to "The international harmonized protocol for the proficiency testing of analytical chemistry laboratories", *Recommendation 1*.

8. Remarks

8.1 Contributions from all accreditation bodies and participants to this programme are gratefully noted. Special thanks are extended to APLAC PT Committee, and their support to the programme.

8.2 If the participants have any queries about this report, please contact the coordinator of this proficiency testing programme as follows:

Guangdong Inspection and Quarantine Technology Center-Toys and Juvenile Products
Testing Lab (IQTC)
Name: Liu Chonghua
Email: liuch@iqtc.cn

China National Accreditation Service for Conformity Assessment (CNAS)
Name: He Ping
Email: heping@cnas.org.cn

8.3 Use of the report other than the demonstration of the performance shall only be allowed with the written permission from APLAC.

9. References

- [1] ISO/IEC 17043:2010, Conformity assessment–General requirements for proficiency testing
- [2] ISO/IEC 17025:2005, General requirements for the competence of testing and calibration laboratories
- [3] ISO 13528: 2015, “Statistical methods for use in proficiency testing by inter-laboratory comparisons”
- [4] BIPM JCGM100, Evaluation of measurement data – Guide to the expression of uncertainty in measurement
- [5] EURACHEM/CITAC Guide CG4, “Quantifying Uncertainty in Analytical Measurement”.
- [6] THOMPSON M., ELLISON S.L.R., WOOD R. “The international Harmonized Protocol for the Proficiency testing of analytical chemistry laboratories” (IUPAC Technical Report). Pure Appl. Chem. 2006, 78 (1) pp. 145-196
- [7] EN71-3:2013+A1:2014, Safety of toys – Migration of certain elements

Table 1 Geographical Distribution of Participants

Economy	Registered Number	Reported Number	Economy	Registered Number	Reported Number
China	6	6	Austria	1	1
Sweden	2	2	France	1	1
Finland	1	1	Philippines	4	4
Italy	2	2	Japan	4	4
Republic of Macedonia	1	1	Republic of Serbia	2	2
USA	5	4	Argentina	1	0
Malaysia	4	4	Australia	1	1
Slovakia	2	2	Portugal	1	1
Canada	2	2	Thailand	4	4
Chinese Taipei	4	4	Korea	1	1
Latvia	1	1	Hong Kong	1	1
Viet Nam	4	4	Mongolia	2	2
Lithuania	1	1	Czech Republic	1	1
Poland	2	2	Turkey	1	1
Denmark	2	1	Indonesia	5	5
India	4	4	Chile	1	0
United Kingdom	2	1	Amman, Jordan	1	1
Morocco	2	2	Tunisia	2	2
KSA	1	1	Iraq	2	2
Sultanate of Oman	2	2			

Table 2 Summary of Participants' Statistical Results (unit:mg/kg)

	Migration of Barium	Migration of Cadmium	Migration of Chromium	Migration of Lead
Number of results	71	78	74	79
x*	298	20.4	25.4	74.6
s*	82.6	6.10	6.16	24.0
CV%	27.7	29.9	24.2	32.1
Max	529.6	73.5	106.38	173.46
Min	142.4	6.475	12.3	30.275
Range	387.2	67.025	94.08	143.185

Note: The x* and s* in this paper are on behalf of robust average and robust standard deviation respectively and were estimated according to ISO 13528:2005. The extreme outliers from lab 008, 058, 072 are not included in the statistical analysis.

Table 3 Summary of Unsatisfactory Participants

	Migration of Barium	Migration of Cadmium	Migration of Chromium	Migration of Lead
Lab code	072	006, 007, 012, 019 , 047, 061, 066, 072	046, 058, 072	007, 008, 012, 027 , 072

Table 4 Summary of Z-scores of Results

z-score	Number of results (Percentage)			
	Migration of Barium	Migration of Cadmium	Migration of Chromium	Migration of Lead
$ z \leq 2.0$	67(94%)	69(88%)	67(91%)	73(92%)
$2.0 < z < 3.0$	4(5.6%)	2(2.6%)	6(8.1%)	3(3.8%)
$ z \geq 3.0$	0(0.0%)	7(9.0%)	1(1.4%)	3(3.8%)

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Total	71(100%)	78(100%)	74(100%)	79(100%)
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Table 5 Summary of Test Method

No.	Method	Number of Lab	Percentage
1	EN 71-3:2013	10	13%
2	EN 71-3:2013+A1:2014	33	42%
3	GB 6675-2014	1	1.3%
4	ISO 8124-3:2010+A1:2014	26	33%
5	ASTM F963-11	5	6.4%
6	Other	3	3.8%

Table 6 Summary of Test Method Used by Participants with Unsatisfactory Results

Method	Migration of Barium	Migration of Cadmium	Migration of Chromium	Migration of Lead
EN 71-3:2013	0	1	1	0
EN 71-3:2013+A1:2014	1	3	1	2
GB 6675-2014	0	0	0	0
ISO 8124-3:2010+A1:2014	0	4	1	1
Other	0	0	0	2

Table 7 Summary of Uncertainty Number

	Migration of Barium	Migration of Cadmium	Migration of Chromium	Migration of Lead
Reported	60	65	63	67
Unreported	12	14	13	14
Percentage	83%	82%	83%	83%

Table 8 Summary of Uncertainty Associated with Assigned Values

k=2	Uncertainty Associated with Assigned Values			
	Migration of Barium	Migration of Cadmium	Migration of Chromium	Migration of Lead
u_x	12.3	0.86	0.89	3.4
$U_x(k=2)$	24.5	1.7	1.8	6.7
$u_{\max}(1.5s^*)$	124	9.1	9.2	36
$U_{\max}(k=2)$	248	18	18	72

Table 9 Summary of the shaking frequency

	The shaking frequency(rpm)				
	<50	50-99	100-150	151-200	>200
participants number	2	18	29	11	5
Percentage	3.1%	28%	45%	17%	7.7%

Table 10 Summary of analysis apparatus

No.	Apparatus	Number of Lab	Percentage
1	ICP-OES	53	69%
2	AAS	12	16%
3	ICP-MS	11	14%
4	other	1	1.3%

Figure 1 Graph of Normality Test for Ba

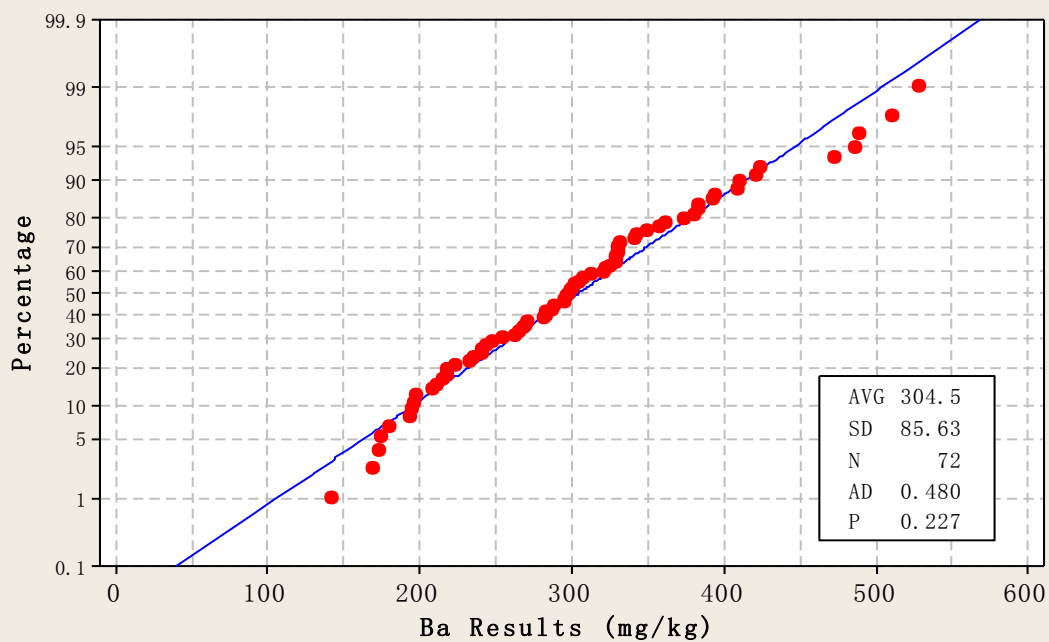
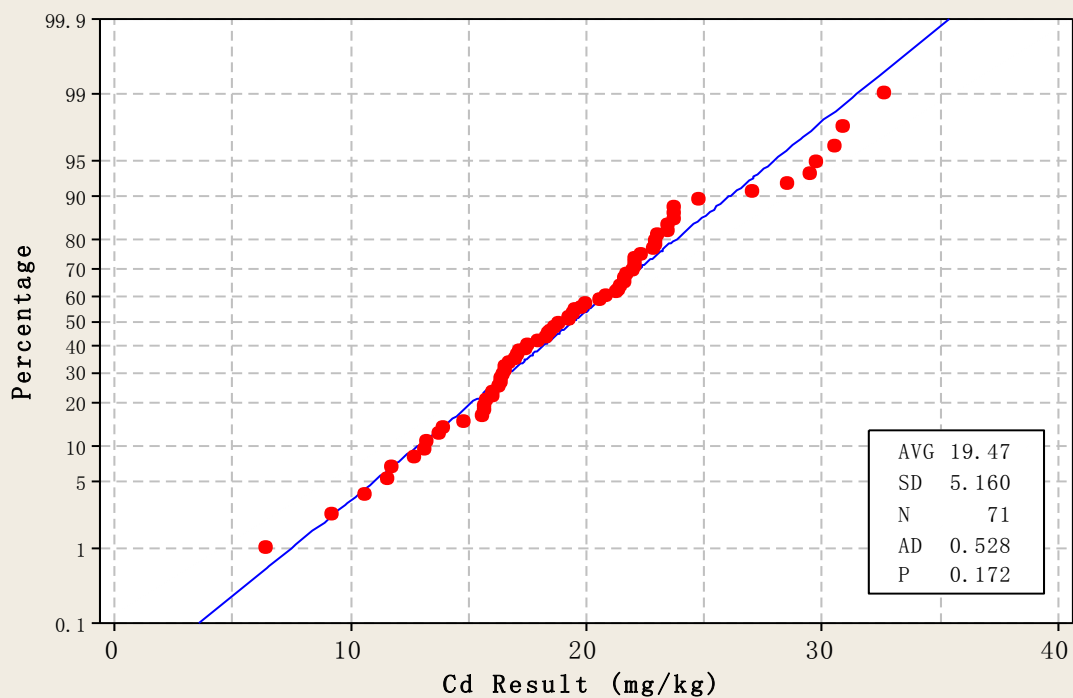
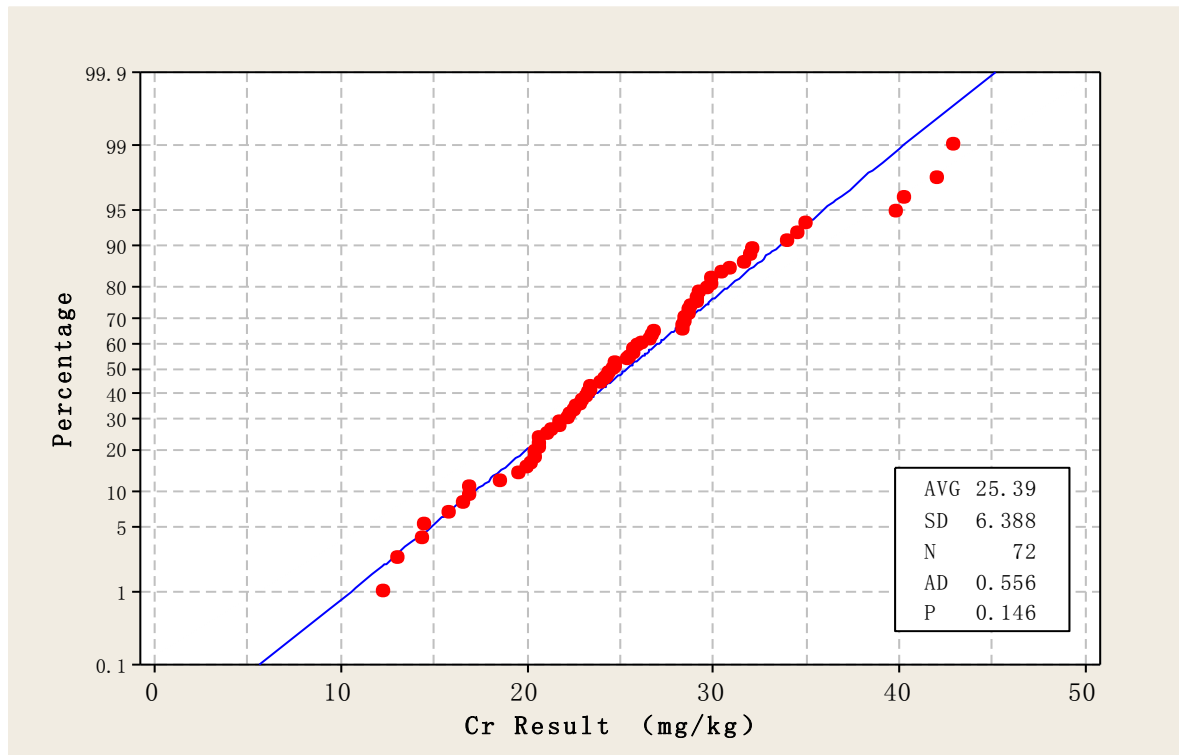


Figure 2 Graph of Normality Test for Cd



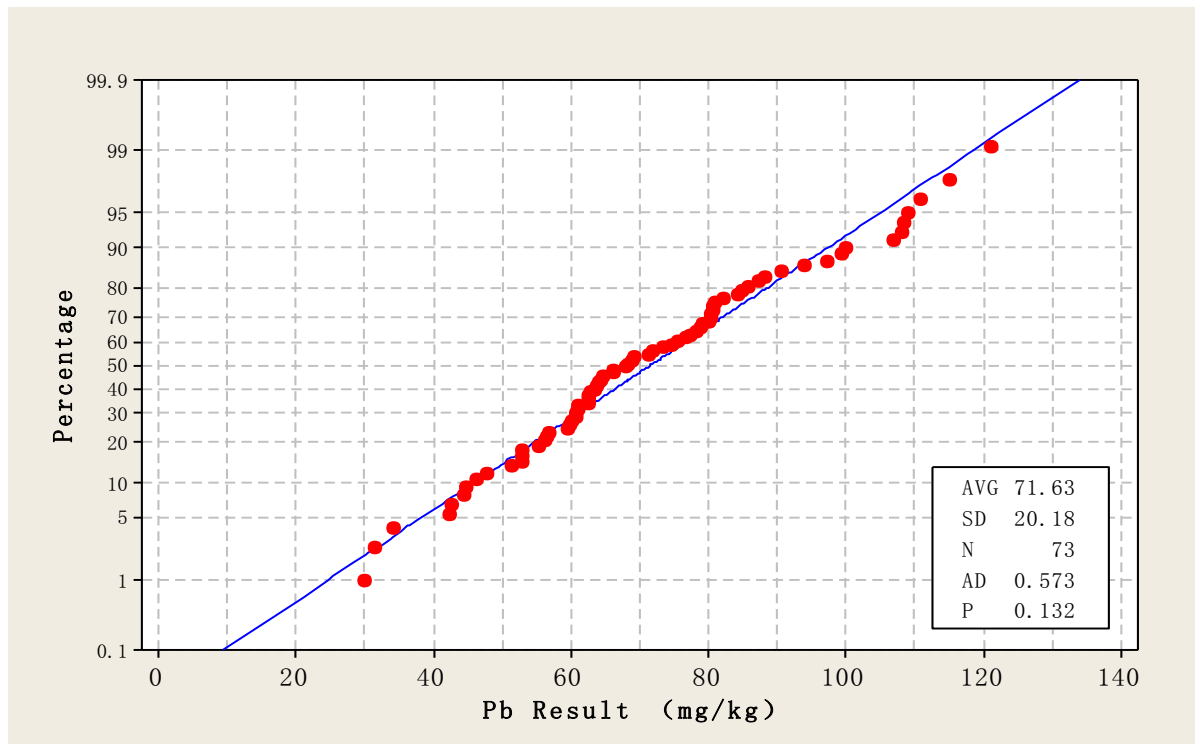
Note: Lab code 006, 007, 012, 019, 047, 061, 066&072 are not included

Figure 3 Graph of Normality Test for Cr



Note: Lab code 007,058,046&072 are not included

Figure 4 Graph of Normality Test for Pb



Note: Lab code 006,007,008,012,019,027,066&072 are not included

Figure 5 Kernel density plot for Ba

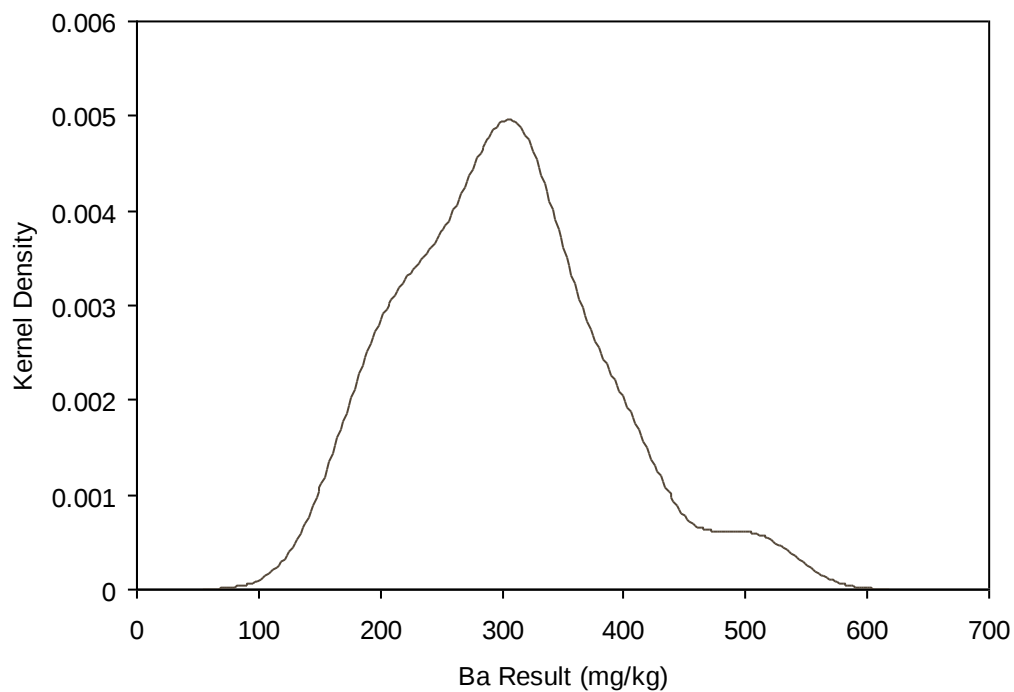


Figure 6 Kernel density plot for Cd

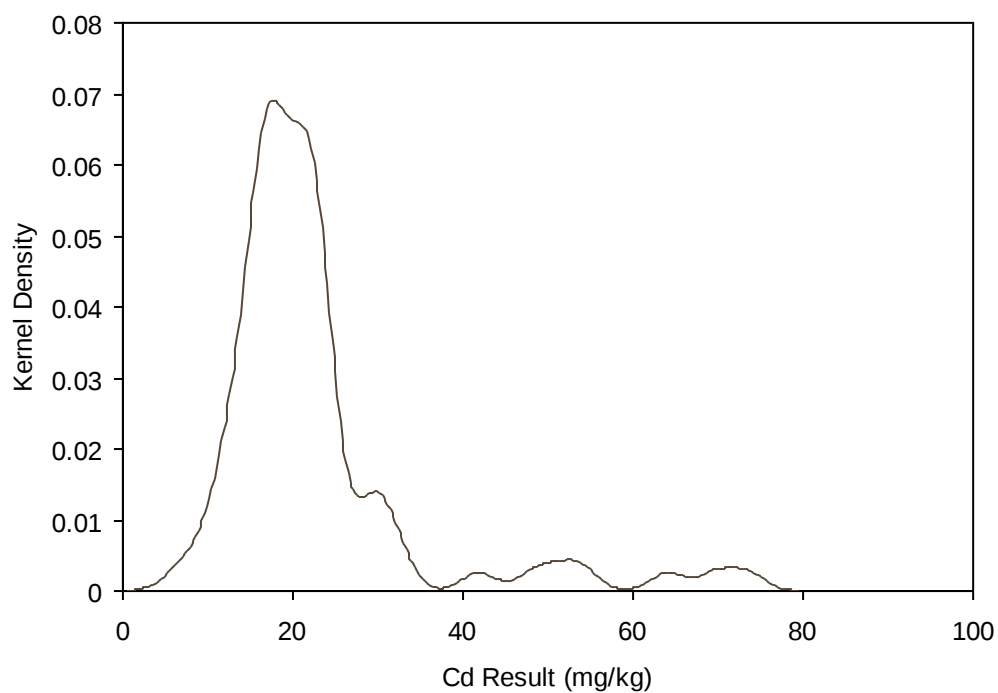


Figure 7 Kernel density plot for Cr

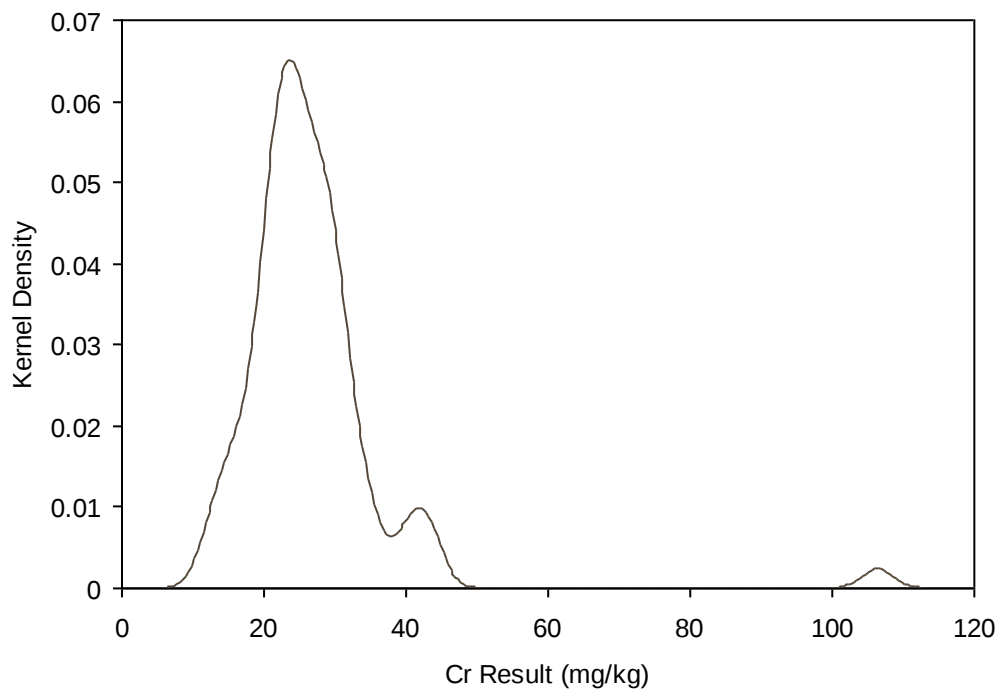


Figure 8 Kernel density plot for Pb

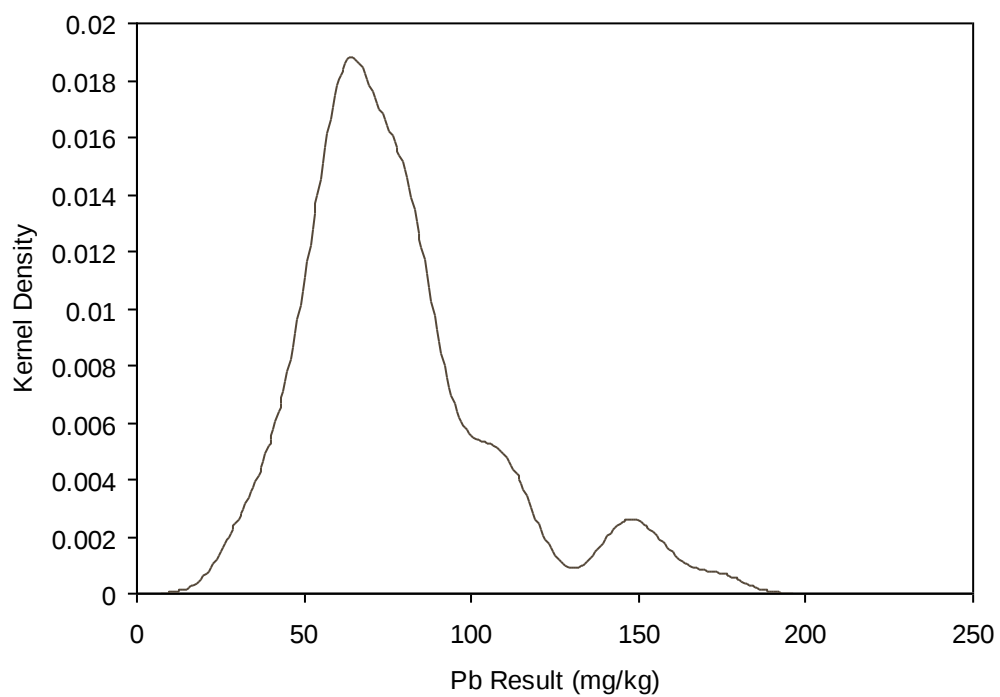
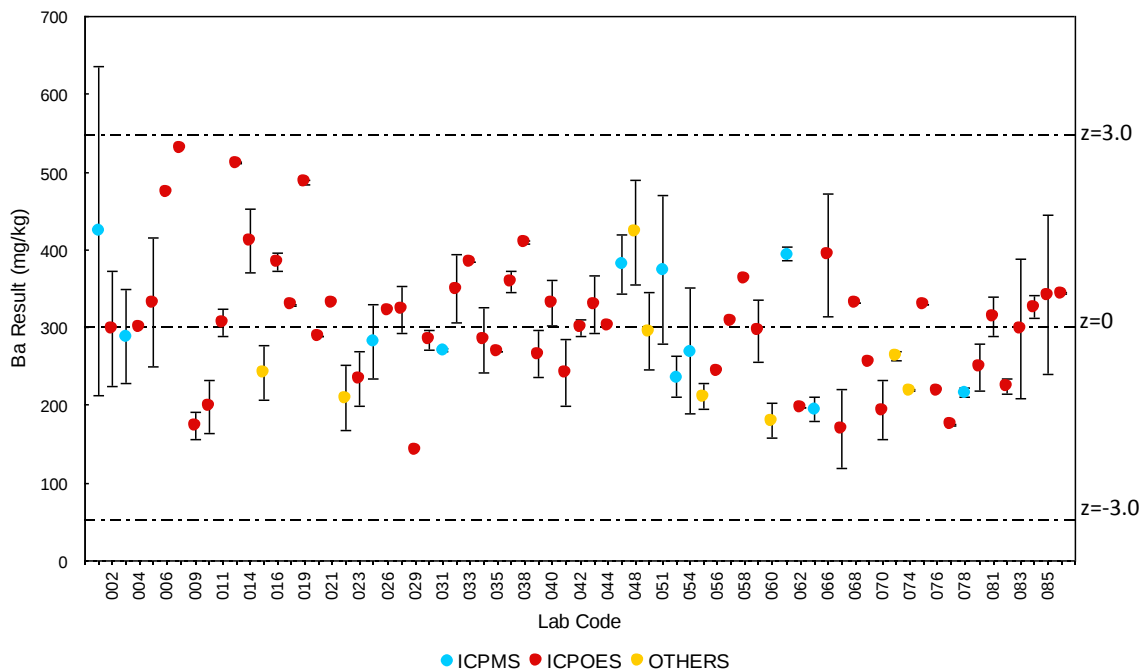
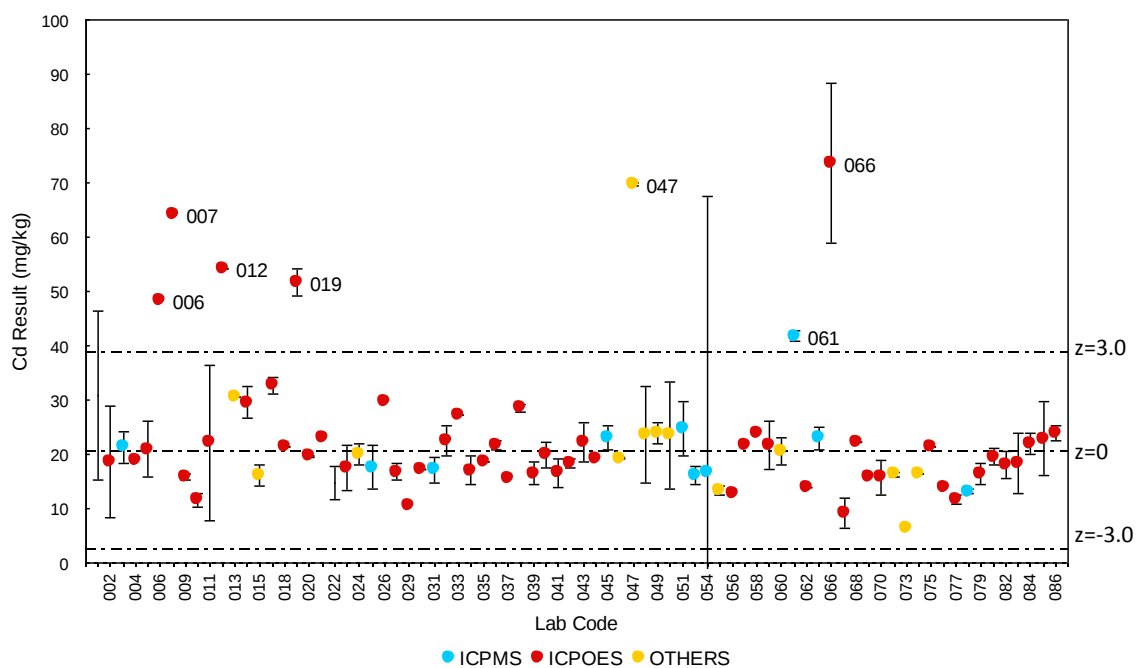


Figure 9 Scatter Diagram for Ba Results with Uncertainty



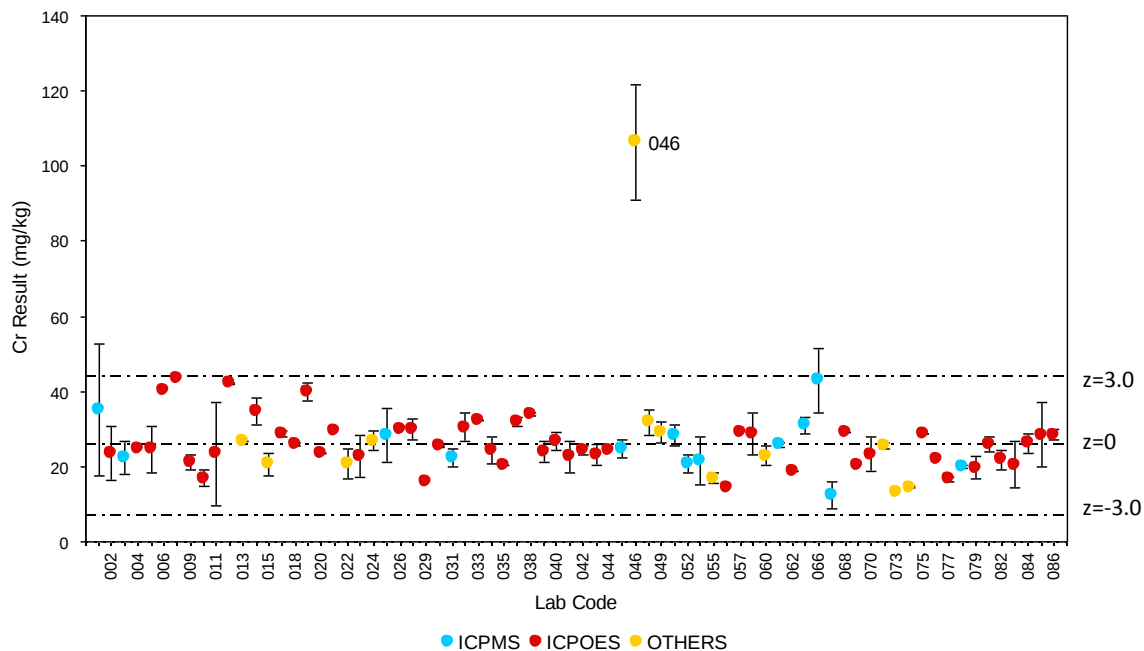
Note: lab code 004,006,007,021,026,029,044,056,057,058,069,076 didn't report uncertainty.

Figure 10 Scatter Diagram for Cd Results with Uncertainty



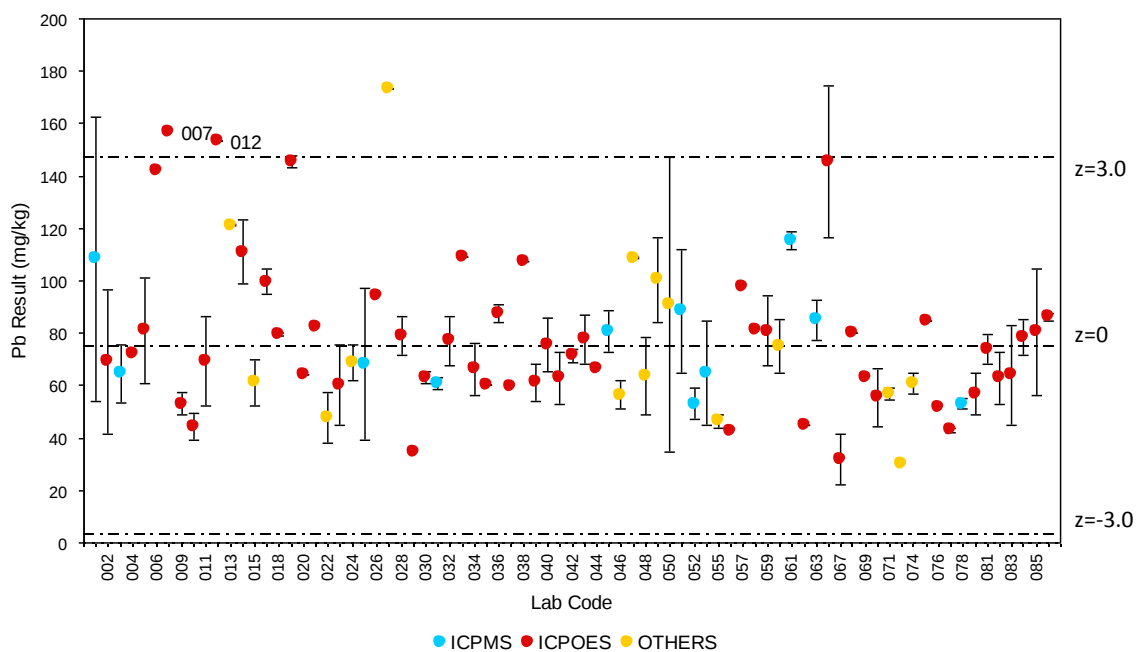
Note: lab code 004,006,007,021,026,029,037,044,056,057,058,069,073,076 didn't report uncertainty.

Figure 11 Scatter Diagram for Cr Results with Uncertainty



Note: lab code 004,006,007,021,026,029,044,056,057,058,069,073,076 didn't report uncertainty.

Figure 12 Scatter Diagram for Pb Results with Uncertainty



Note: lab code 004,006,007,021,026,029,037,044,056,057,058,069,073,076 didn't report uncertainty.

APPENDIX A Results and Statistical Analysis

Table A.1 Results of Barium

Lab code	Accreditation status	Uncertainty	Result 1 (mg/kg)	Result 2 (mg/kg)	Reported Average Result (mg/kg)	z-score
001	Yes	212.2	453.7	395.1	424.4	1.53
002	Yes	25%(rel.)	301.0	294.8	297.9	0.00
003	Yes	61	291.0	286.0	288.5	-0.11
004	Yes	--	306.2	296.2	301.2	0.04
005	Yes	82.9	329.8	333.4	331.6	0.41
006	Yes	--	481.4	466.3	473.9	2.13
007	Yes	--	492.3	566.8	529.6	2.80
008	Yes	--	--	--	--	--
009	Yes	17.4	--	--	174.2	-1.50
010	Yes	34.4	202.1	195.0	198.6	-1.20
011	Yes	18.1	305.9	305.9	305.9	0.10
012	Yes	±0.08	504.0	519.5	511.8	2.59
013	No	--	--	--	--	--
014	Yes	41.1	381.6	441.1	411.4	1.37
015	Yes	34.6	245.2	239.1	242.1	-0.68
016	Yes	11.5	372.1	396.1	384.1	1.04
017	Yes	--	--	--	--	--
018	Yes	0.79	329.3	328.7	329.3	0.38
019	Yes	±2.5	488.5	485.3	486.9	2.29
020	Yes	0.0517	298.1	278.5	288.3	-0.12
021	Yes	--	322.8	341.1	332.0	0.41
022	Yes	42	204.6	213.0	208.8	-1.08
023	Yes	35.2	223.6	243.5	233.6	-0.78
024	Yes	--	--	--	--	--
025	Yes	47.6	298.7	265.3	282.0	-0.19
026	Yes	--	325.9	317.7	321.8	0.29
027	No	--	--	--	--	--
028	Yes	30.2	324.8	322.3	323.6	0.31
029	Yes	--	141.8	142.9	142.4	-1.88
030	Yes	12.7	289.9	287.4	284.1	-0.17
031	Yes	2.4	272.1	271.0	271.5	-0.32
032	Yes	12.4%	350.1	349.1	349.6	0.63
033	Yes	0.0226	380.4	388.7	384.6	1.05
034	Yes	±42.6	280.7	287.3	284.0	-0.17
035	Yes	0.00985	270.0	270.0	270.0	-0.34
036	--	13.6	360.7	355.9	358.3	0.73
037	No	--	--	--	--	--
038	No	1.5	409.2	410.2	409.7	1.35
039	Yes	30.8	266.8	265.5	266.1	-0.39
040	Yes	29.4	338.6	332.8	330.7	0.40

APLAC T098 Determination of Migration of Heavy Metals in Toys Coating

Lab code	Accreditation status	Uncertainty	Result 1 (mg/kg)	Result 2 (mg/kg)	Reported Average Result (mg/kg)	z-score
041	Yes	±42.6	243.7	240.1	241.9	-0.68
042	Yes	11.2	293.7	305.1	299.4	0.02
043	Yes	37.2	344.3	314.6	329.5	0.38
044	Yes	--	312.5	293.5	303	0.06
045	Yes	38	384.1	379.5	381.8	1.02
046	No	--	--	--	--	--
047	No	--	--	--	--	--
048	No	66.93	--	--	422.3	1.51
049	Yes	50.2	297.0	293.6	295.3	-0.03
050	Yes	--	--	--	--	--
051	Yes	96.2	376.5	372.5	374.5	0.93
052	Yes	25.99	232.5	240	236.3	-0.75
053	Yes	--	--	--	--	--
054	Yes	81	278.9	259.6	269.3	-0.35
055	Yes	16.78	--	--	211.27	-1.05
056	Yes	--	243.5	244.5	244.0	-0.65
057	Yes	--	292.6317	324.6667	308.6492	0.13
058	Yes	--	358.1	367.6	362.9	0.79
059	Yes	±40.2	308.7	282.8	295.8	-0.03
060	Yes	22.84	182.9	177.4	180.2	-1.43
061	Yes	2.2 at 98.16	--	--	394.3279	1.17
062	Yes	0.043	195.9	198.2	197.0	-1.22
063	Yes	3 at 38mg/kg	194	196	195	-1.25
064	Yes	--	--	--	--	--
065	Yes	--	--	--	--	--
066	Yes	78.7	365	422	393.5	1.16
067	Yes	50.82	179.2	159.6	169.4	-1.56
068	Yes	0.1375	330.3	331.5	330.9	0.40
069	Yes	--	252.8	258.0	255.4	-0.52
070	No	38.8	194.0	194.0	194.0	-1.26
071	No	6.2	267.6	258.7	263.1	-0.42
072	No	10.6	490.2	490.4	490.3	action
073	Yes	--	--	--	--	--
074	Yes	1.1	219.65	218.55	219.1	-0.95
075	No	0.004	327.2	331.8	329.5	0.38
076	No	--	--	--	218.7	-0.96
077	Yes	0.66	--	--	174.9	-1.49
078	Yes	5.033	--	--	216.3	-0.99
079	Yes	30	273.3	224.7	249.0	-0.59
080	--	--	--	--	--	--
081	Yes	±25.1	311.2	316.3	313.7	0.19
082	Yes	10	222.0	227.1	224.6	-0.89

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Lab code	Accreditation status	Uncertainty	Result 1 (mg/kg)	Result 2 (mg/kg)	Reported Average Result (mg/kg)	z-score
083	Yes	89.6(K=2)	299.4	297.7	298.6	0.01
084	Yes	14.6	326.4	325.6	326.0	0.34
085	Yes	102.5	344.9	338.7	341.8	0.53
086	Yes	1.4	346.2	341.5	343.8	0.55

Table A.2 Results of Cadmium

Lab code	Accreditation status	Uncertainty	Result 1 (mg/kg)	Result 2 (mg/kg)	Reported Average Result (mg/kg)	z-score
001	Yes	15.5	33.3	28.4	30.9	1.72
002	Yes	55%(rel.)	19.1	18.2	18.7	-0.28
003	Yes	3	21.4	21.1	21.3	0.15
004	Yes	--	18.4	19.3	18.9	-0.25
005	Yes	5.2	20.5	21.2	20.9	0.08
006	Yes	--	57.15	39.46	48.3	4.58
007	Yes	--	61.8	66.4	64.1	7.17
008	Yes	--	--	--	--	--
009	Yes	0.58	--	--	15.77	-0.76
010	Yes	1.3	11.8	11.3	11.6	-1.44
011	Yes	14.4	22.3	21.8	22.1	0.28
012	Yes	±0.06	49.5	58.9	54.2	5.55
013	No	0.000962	29.6	31.7	30.6	1.67
014	Yes	2.9	29.8	29.2	29.5	1.49
015	Yes	2.0	15.8	16.4	16.1	-0.71
016	Yes	1.6	31.9	33.5	32.7	2.02
017	Yes	--	--	--	--	--
018	Yes	0.04	21.4	21.3	21.4	0.16
019	Yes	±2.5	52.0	51.2	51.6	5.12
020	Yes	0.0421	20.0	19.2	19.6	-0.13
021	Yes	--	22.4	23.5	23.0	0.43
022	Yes	3.0	14.4	15.2	14.8	-0.92
023	Yes	4.2	16.3	18.7	17.5	-0.48
024	Yes	2.0	19.2	21.0	20.0	-0.07
025	Yes	4.00	18.9	16.2	17.55	-0.47
026	Yes	--	31.52	28.02	29.77	1.54
027	No	--	--	--	--	--
028	Yes	1.6	17.1	16.8	16.8	-0.59
029	Yes	--	10.5	10.6	10.6	-1.61
030	Yes	0.1	17.4	16.9	17.2	-0.52
031	Yes	2.4	16.9	17.3	17.1	-0.54
032	Yes	12.4%	21.6	23.1	22.4	0.33

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Lab code	Accreditation status	Uncertainty	Result 1 (mg/kg)	Result 2 (mg/kg)	Reported Average Result (mg/kg)	z-score
033	Yes	0.0092	26.4	27.7	27.1	1.10
034	Yes	±2.6	16.8	17.2	17.0	-0.56
035	Yes	0.00153	18.5	18.5	18.5	-0.31
036	--	0.8	21.9	21.6	21.7	0.21
037	Yes	--	16.0	15.2	15.6	-0.79
038	Yes	0.7	28.3	28.8	28.6	1.35
039	Yes	2.0	16.4	16.5	16.5	-0.64
040	Yes	2.4	21.0	18.8	19.9	-0.08
041	Yes	±2.7	16.2	17.0	16.6	-0.62
042	Yes	0.8	18.0	18.5	18.3	-0.34
043	Yes	3.6	21.8	22.4	22.1	0.28
044	Yes	--	20.0	18.5	19.3	-0.18
045	Yes	2.3	23.1	23.1	23.1	0.44
046	Yes	0.16	--	--	19.28	-0.18
047	No	0.34	69.9	69.5	69.7	8.09
048	No	8.876	--	--	23.50	0.51
049	Yes	1.9	23.5	24.1	23.8	0.56
050	Yes	Res ₁ :6.1/ Res ₂ :7.6 1)	23.2	23.8	23.5	0.51
051	Yes	5.0	25.0	24.7	24.8	0.72
052	Yes	1.77	16.4	15.7	16.1	-0.71
053	Yes	--	--	--	--	--
054	Yes	51	17.1	16.1	16.6	-0.62
055	Yes	0.89	--	--	13.26	-1.17
056	Yes	--	12.6	12.7	12.7	-1.26
057	Yes	--	22.0350	21.4700	21.7525	0.22
058	Yes	--	23.6	23.9	23.8	0.56
059	Yes	±4.4	23.3	20.0	21.7	0.21
060	Yes	2.56	21.0	20.2	20.6	0.03
061	Yes	2.3 at 98.56	--	--	41.8026	3.51
062	Yes	0.026	13.3	14.7	14.0	-1.05
063	Yes	2.8 at 31mg/kg	21	25	23	0.43
064	Yes	--	--	--	--	--
065	Yes	--	--	--	--	--
066	Yes	14.7	82	65	73.5	8.71
067	Yes	2.78	9.73	8.77	9.25	-1.83
068	Yes	0.00006	22.1	22.1	22.1	0.28
069	Yes	--	15.5	15.8	15.7	-0.77
070	No	3.1	15.7	15.7	15.7	-0.77
071	No	0.5	16.7	16.0	16.3	-0.67
072	No	10.2	131.1	131.3	131.2	action
073	Yes	--	7.35	5.6	6.475	-2.28

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Lab code	Accreditation status	Uncertainty	Result 1 (mg/kg)	Result 2 (mg/kg)	Reported Average Result (mg/kg)	z-score
074	Yes	0.13	16.46	16.33	16.39	-0.66
075	No	0.004	21.0	22.0	21.5	0.18
076	No	--	--	--	13.81	-1.08
077	Yes	0.83	--	--	11.79	-1.41
078	Yes	0.5033	--	--	13.17	-1.19
079	Yes	2	17.9	14.9	16.4	-0.66
080	--	--	--	--	--	--
081	Yes	±1.5	19.4	19.5	19.5	-0.15
082	Yes	2.5	18.1	17.9	18.0	-0.39
083	Yes	5.5(K=2)	18.2	18.6	18.4	-0.33
084	Yes	1.9	22.0	22.0	22.0	0.26
085	Yes	6.8	23.05	22.71	22.9	0.41
086	Yes	1.4	24.6	23.1	23.8	0.56

Table A.3 Results of Chromium

Lab code	Accreditation status	Uncertainty	Result 1 (mg/kg)	Result 2 (mg/kg)	Reported Average Result (mg/kg)	z-score
001	Yes	17.5	38.3	31.7	35.0	1.56
002	Yes	30%(rel.)	24.4	22.5	23.5	-0.31
003	Yes	4.5	22.8	21.7	22.3	-0.51
004	Yes	--	24.5	25.0	24.8	-0.10
005	Yes	6.2	24.4	24.9	24.7	-0.12
006	Yes	--	42.18	38.41	40.3	2.42
007	Yes	--	41.6	45.4	43.5	2.94
008	Yes	--	--	--	--	--
009	Yes	2.01	--	--	21.10	-0.70
010	Yes	2.3	17.5	16.2	16.9	-1.38
011	Yes	13.7	23.0	23.7	23.4	-0.33
012	Yes	±0.07	41.5	42.7	42.1	2.71
013	no	0.00054	28.1	25.2	26.6	0.19
014	Yes	3.6	31.1	38.2	34.6	1.49
015	Yes	3.0	21.7	19.6	20.7	-0.77
016	Yes	0.9	29.0	28.6	28.8	0.55
017	Yes	--	--	--	--	--
018	Yes	0.32	25.9	26.0	26.0	0.09
019	Yes	±2.5	40.3	39.5	39.9	2.35
020	Yes	0.0728	24.3	22.7	23.5	-0.31
021	Yes	--	29.2	30.1	29.7	0.70
022	Yes	4.1	20.5	21.0	20.7	-0.77
023	Yes	5.6	21.0	24.2	22.6	-0.46

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Lab code	Accreditation status	Uncertainty	Result 1 (mg/kg)	Result 2 (mg/kg)	Reported Average Result (mg/kg)	z-score
024	Yes	2.7	26.7	27.0	26.9	0.24
025	Yes	7.26	29.7	27.2	28.4	0.48
026	Yes	--	30.89	29.10	29.99	0.74
027	No	--	--	--	--	--
028	Yes	2.8	30.4	29.7	30.0	0.74
029	Yes	--	15.4	16.3	15.8	-1.56
030	Yes	0.2	25.5	25.5	25.5	0.01
031	Yes	2.4	21.9	22.4	22.2	-0.52
032	Yes	12.3%	30.4	30.6	30.5	0.83
033	Yes	0.0164	31.7	32.7	32.2	1.10
034	Yes	±3.6	24.1	24.3	24.2	-0.20
035	Yes	0.00120	20.5	20.5	20.5	-0.80
036	--	1.2	31.9	32.0	32.0	1.07
037	No	--	--	--	--	--
038	Yes	0.3	34.1	33.9	34.0	1.39
039	Yes	2.8	24.7	23.3	24.0	-0.23
040	Yes	2.3	27.8	25.6	26.7	0.21
041	Yes	±4.2	22.9	22.4	22.7	-0.44
042	Yes	1.3	23.5	25.0	24.3	-0.18
043	Yes	2.8	22.8	23.1	23.0	-0.39
044	Yes	--	25.0	24.0	24.5	-0.15
045	Yes	2.5	24.7	25.0	24.8	-0.10
046	No	15.27	--	--	106.38	13.15
047	No	--	--	--	--	--
048	No	3.458	--	--	31.76	1.03
049	Yes	2.9	29.3	29.1	29.2	0.61
050	Yes	--	--	--	--	--
051	Yes	2.7	28.0	28.7	28.4	0.48
052	Yes	2.28	20.7	20.8	20.7	-0.77
053	Yes	--	--	--	--	--
054	Yes	6.4	22.8	20.8	21.4	-0.65
055	Yes	1.41	--	--	16.90	-1.38
056	Yes	--	14.3	14.5	14.4	-1.79
057	Yes	--	31.1170	27.2700	29.1935	0.61
058	Yes	--	26.9	29.3	56.2	action
059	Yes	±5.6	30.1	27.8	28.9	0.57
060	Yes	2.71	22.2	23.6	22.9	-0.41
061	Yes	2.1 at 98.82	--	--	25.7873	0.06
062	Yes	0.035	17.8	19.4	18.6	-1.11
063	Yes	2.5 at 36mg/kg	30	31	31	0.91
064	Yes	--	--	--	--	--
065	Yes	--	--	--	--	--

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Lab code	Accreditation status	Uncertainty	Result 1 (mg/kg)	Result 2 (mg/kg)	Reported Average Result (mg/kg)	z-score
066	Yes	8.6	47	39	43	2.86
067	Yes	3.69	12.91	11.69	12.30	-2.13
068	Yes	0.00463	29.2	29.4	29.3	0.63
069	Yes	--	20.0	20.5	20.3	-0.83
070	No	4.6	23.2	23.2	23.2	-0.36
071	No	0.8	26.0	24.8	25.4	0.00
072	No	10.4	290.5	290.7	290.6	action
073	Yes	--	14.35	11.9	13.125	-2.00
074	Yes	0.13	14.46	14.59	14.52	-1.77
075	No	0.004	28.8	28.8	28.8	0.55
076	No	--	--	--	21.75	-0.60
077	Yes	0.59	--	--	16.67	-1.42
078	Yes	0.5033	--	--	20.03	-0.87
079	Yes	3	20.9	18.4	19.6	-0.94
080	--	--	--	--	--	--
081	Yes	±2.0	25.8	25.8	25.8	0.06
082	Yes	2.5	21.8	21.9	21.8	-0.59
083	Yes	6.1(K=2)	20.3	20.6	20.5	-0.80
084	Yes	2.6	26.1	26.2	26.2	0.13
085	Yes	8.6	28.00	28.98	28.5	0.50
086	Yes	1.4	29.2	27.9	28.5	0.50

Table A.4 Results of Lead

Lab code	Accreditation status	Uncertainty	Result 1 (mg/kg)	Result 2 (mg/kg)	Reported Average Result (mg/kg)	z-score
001	Yes	54.2	120.6	96.0	108.3	1.40
002	Yes	40%(rel.)	69.5	68.6	69.1	-0.23
003	Yes	11	64.8	64.2	64.5	-0.42
004	Yes	--	73.2	71.0	72.1	-0.11
005	Yes	20.2	81.7	80.3	81.0	0.27
006	Yes	--	147.5	136.8	142.2	2.82
007	Yes	--	155.2	158.2	156.7	3.42
008	Yes	44.8	249.7	258.8	254.2	action
009	Yes	4.31	--	--	53.07	-0.90
010	Yes	5.1	45.5	43.4	44.5	-1.26
011	Yes	17.2	69.7	69.1	69.4	-0.22
012	Yes	±0.13	150.7	156.3	153.5	3.29
013	no	0.019926	121.4	121.3	121.3	1.95
014	Yes	12.2	110.7	111.3	111.0	1.52
015	Yes	8.8	61.3	61.2	61.2	-0.56

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Lab code	Accreditation status	Uncertainty	Result 1 (mg/kg)	Result 2 (mg/kg)	Reported Average Result (mg/kg)	z-score
016	Yes	5.0	100.2	99.2	99.7	1.05
017	Yes	--	--	--	--	--
018	Yes	0.33	79.4	79.6	79.5	0.20
019	Yes	±2.5	144.9	145.9	145.4	2.95
020	Yes	0.0444	67.0	61.9	64.4	-0.43
021	Yes	--	78.8	85.7	82.3	0.32
022	Yes	9.6	46.9	48.9	47.9	-1.11
023	Yes	15.4	55.9	64.6	60.3	-0.60
024	Yes	6.9	69.3	67.9	68.6	-0.25
025	Yes	29.01	77.4	59.0	68.2	-0.27
026	Yes	--	97.61	90.88	94.24	0.82
027	No	0.017%	174.66	172.25	173.46	4.12
028	Yes	7.4	80.5	77.9	79.2	0.19
029	Yes	--	33.5	35.5	34.5	-1.67
030	Yes	2.3	62.2	63.8	63.0	-0.49
031	Yes	2.4	59.7	62.1	60.9	-0.57
032	Yes	12.4%	74.9	79.0	77.0	0.10
033	Yes	0.0324	106.4	111.9	109.2	1.44
034	Yes	±10.0	65.6	67.0	66.3	-0.35
035	Yes	0.00215	60.0	60.0	60.0	-0.61
036	--	3.3	88.1	87.1	87.6	0.54
037	Yes	--	61.7	58.0	59.8	-0.62
038	Yes	0.1	107.3	107.3	107.3	1.36
039	Yes	7.1	61.8	60.6	61.2	-0.56
040	Yes	10.3	79.1	72.3	75.7	0.04
041	Yes	±9.9	61.4	64.1	62.8	-0.49
042	Yes	2.7	70.2	72.5	71.4	-0.13
043	Yes	9.4	77.0	78.1	77.6	0.12
044	Yes	--	68.0	64.5	66.3	-0.35
045	Yes	8.0	79.9	81.3	80.6	0.25
046	Yes	5.46	--	--	56.48	-0.76
047	Yes	0.4	108.9	108.5	108.7	1.42
048	No	14.97	-	-	63.67	-0.46
049	Yes	16.0	99.0	101.7	100.3	1.07
050	Yes	Res ₁ :35.0/ Res ₂ :44.3 1)	89.1	92.7	90.9	0.68
051	Yes	23.4	88.2	88.6	88.4	0.57
052	Yes	5.83	53.7	52.2	53	-0.90
053	Yes	--	--	--	--	--
054	Yes	20	67.7	62.2	64.9	-0.41
055	Yes	2.49	--	--	46.51	-1.17
056	Yes	--	42.6	42.6	42.6	-1.34

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Lab code	Accreditation status	Uncertainty	Result 1 (mg/kg)	Result 2 (mg/kg)	Reported Average Result (mg/kg)	z-score
057	Yes	--	87.3550	107.5365	97.4458	0.95
058	Yes	--	83.7	78.8	81.3	0.28
059	Yes	±13.3	85.4	76.1	80.8	0.26
060	Yes	10.17	77.9	71.7	74.8	0.01
061	Yes	2.9 at 98.66	--	--	115.3092	1.70
062	Yes	0.082	44.8	45.2	45.0	-1.24
063	Yes	3 at 33mg/kg	90	80	85	0.43
064	Yes	--	--	--	--	--
065	Yes	--	--	--	--	--
066	Yes	29.1	152	139	145.5	2.96
067	Yes	9.51	33.56	29.82	31.69	-1.79
068	Yes	0.00105	80.3	80.2	80.15	0.23
069	Yes	--	62.0	63.5	62.8	-0.49
070	No	11.1	55.5	55.5	55.5	-0.80
071	No	2.3	58.6	55.4	57.0	-0.74
072	No	10.1	155.3	155.1	155.2	action
073	Yes	--	26.95	33.6	30.275	-1.85
074	Yes	4.02	59.02	63.04	61.03	-0.57
075	No	0.003	85.6	83.1	84.4	0.41
076	No	--	--	--	51.65	-0.96
077	Yes	1.03	--	--	42.90	-1.32
078	Yes	1.780	--	--	53.10	-0.90
079	Yes	8	62.4	51.3	56.8	-0.74
080	--	--	--	--	--	--
081	Yes	±5.6	74.7	72.6	73.7	-0.04
082	Yes	10	61.9	63.6	62.8	-0.49
083	Yes	19.2(K=2)	64.7	63.2	64.0	-0.44
084	Yes	6.9	78.3	78.6	78.4	0.16
085	Yes	24.2	80.10	81.00	80.6	0.25
086	Yes	1.4	82.4	89.8	86.1	0.48

Note1: "--"Results or information were not provided.

Note2: Red color means Unsatisfactory while green means Questionable. "action" means the lab receive an action signal.

Table A.5 Review of Test Methods

Lab Code	Testing standard	Volume (mL)	Sample Weight (g)	Acid Volume (mL)	Shaking frequency (rpm)	Testing instrument
001	EN 71-3:2013+A1:2014	60	0.1409/ 0.1648	7.1/8.2	--	ICP-MS
002	EN 71-3:2013+A1:2014	20	0.20	10	200	ICP-OES
003	EN 71-3:2013+A1:2014	--	0.5	25	85	ICP-MS
004	EN 71-3:2013+A1:2014 /ASTM F963-11	25	0.1148	5.7	150	ICP-OES
005	EN 71-3:2013+A1:2014	50	0.150	7.5	105	ICP-OES
006	ISO 8124-3:2010+A1:2014	250	1	50	150	ICP-OES
007	ISO 8124-3:2010+A1:2014	150	0.5035/ 0.5029	25.20/25.10	140	ICP-OES
008	others-CPSC-CH-E1003-09 /ASTM E1413	--	0.1000	20	N/A	FAAS
009	EN 71-3:2013	5	0.01	~5	--	ICP-OES
010	ASTM F963-11	12.5	0.25	12.5	120	ICP-OES
011	EN 71-3:2013+A1:2014	20	0.3024	15	70	ICP-OES
012	EN 71-3:2013+A1:2014	50	0.10	5.0	120	ICP-OES
013	ISO 8124-3:2010+A1:2014	50	0.5	25	2400	GF-AAS
014	EN 71-3:2013+A1:2014	100	0.5146	25	60	ICP-OES
015	EN 71-3:2013	100	0.5	25	--	FAAS/ICP-MS
016	GB 6675-2014	25	0.2	10	110	ICP-OES
017	--	--	--	--	--	--
018	ISO 8124-3:2010+A1:2014	56	1.0000	56	-	ICP-OES
019	ISO 8124-3:2010+A1:2014	50	1	50	-	ICP-OES
020	Others MS ISO 8124-3:2012	50	±0.3	±15	90	ICP-OES
021	EN 71-3:2013+A1:2014	25	0.0346	5	170	ICP-OES
022	ISO 8124-3:2010+A1:2014	50	0.5	25	20	FAAS/ICP-OES/ICPMS/Others

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Lab Code	Testing standard	Volume (mL)	Sample Weight (g)	Acid Volume (mL)	Shaking frequency (rpm)	Testing instrument
023	EN 71-3:2013+A1:2014	100	0.5	25	60-80	ICP-OES
024	EN 71-3:2013+A1:2014	100	0.5	25	50	FAAS
025	EN 71-3:2013+A1:2014	50	0.2	10.0	60	ICP-MS
026	ASTM F963-11/ISO 8124-3:2010+A1:2014/EN 71-3:2013+A1:2014	12	0.119	6.0	65	ICP-OES
027	--	--	--	--	--	--
028	EN 71-3:2013+A1:2014	25	0.2002	10	170	ICP-OES
029	EN 71-3:2013+A1:2014	15	0.2000	10	180	ICP-OES
030	ISO 8124-3:2010+A1:2014	125	0.3	15	-	ICP-OES
031	EN 71-3:2013+A1:2014	50	~0.2xxx	10	-	ICP-MS
032	ASTM F963-11	--	--	--	170	ICP-OES
033	ASTM F963-11	250	0.4043/ 0.4111	20/21	100	ICP-OES
034	EN 71-3:2013+A1:2014	50	0.2	10	60	ICP-OES
035	EN 71-3:2013+A1:2014	100	0.5	25	110	ICP-OES
036	ISO 8124-3:2010+A1:2014	50	0.2	10	100	ICP-OES
037	ISO 8124-3:2010+A1:2014	20	0.1	5	60	ICP-OES
038	EN 71-3:2013+A1:2014	10	0.2	0.0579	60	ICP-OES
039	ISO 8124-3:2010+A1:2014	10	0.1000	5	110	ICP-OES
040	EN 71-3:2013	100	0.05	25	100	ICP-OES
041	ISO 8124-3:2010+A1:2014	25	0.1	5	140±10	ICP-OES
042	EN 71-3:2013+A1:2014	50	0.5	25	300	ICP-OES
043	EN 71-3:2013+A1:2014	25	0.1	5	60	ICP-OES
044	EN 71-3:2013+A1:2014	60	0.5005	25	150	ICP-OES
045	ISO 8124-3:2010+A1:2014/EN 71-3:2013+A1:2014	40	0.1331/ 0.1460	6.66/7.30	170±20	ICP-MS
046	EN 71-3:2013	50	0.4010	50	150	Others, GFASSS
047	ISO 8124-3:2010+A1:2014	20	0.09	5	50	FAAS

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Lab Code	Testing standard	Volume (mL)	Sample Weight (g)	Acid Volume (mL)	Shaking frequency (rpm)	Testing instrument
048	EN 71-3:2013	100	0.3005	15	-	ET-AAS
049	EN 71-3:2013+A1:2014	50	0.5	25	120	FAAS
050	Others EN 71-3:2013 Category III:Scraped-off	100	1.1087/ 0.8864	55,8513g /44,9485g(weigh t od added c(HCl) = 0,07 mol/L)	no data available	FAAS
051	EN 71-3:2013+A1:2014	100	0.5	25	110	ICP-MS
052	EN 71-3:2013+A1:2014	50	0.1023/ 0.1030	5	-	ICP-MS
053	--	--	--	--	--	--
054	EN 71-3:2013+A1:2014	50	0.2	10	60	ICP-MS
055	--	--	--	--	--	--
056	ISO 8124-3:2010+A1:2014	100	0.5068	25	120	ICP-OES
057	EN 71-3:2013+A1:2014	25	0.1	5	170	ICP-OES
058	ISO 8124-3:2010+A1:2014	20	0.1006	5	170	ICP-OES
059	EN 71-3:2013+A1:2014	25	0.1000	5	~150	ICP-OES
060	EN 71-3:2013+A1:2014	50	0.1	5	150	FAAS/ Others
061	EN 71-3:2013	50	0.2031	10	150	ICP-MS
062	EN 71-3:2013+A1:2014	50	0.30	15	170	ICP-OES
063	EN 71-3:2013	100	0.2	10	60	ICP-MS
064	--	--	--	--	--	--
065	--	--	--	--	--	--
066	EN 71-3:2013+A1:2014	26	--	--	Equipment does not have rpm display. Test method does not specifs rpm requirments	ICP-OES
067	En 71-3:1994 and BS EN 71-3:1995	50	0.50	25	200	ICP-OES
068	EN 71-3:2013+A1:2014	300	0.3	300	400	ICP-OES
069	Others NM EN71-3:2014	100	0.5	25	100	ICP-OES
070	EN 71-3:2013+A1:2014	150	0.8	40	200	ICP-OES

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Lab Code	Testing standard	Volume (mL)	Sample Weight (g)	Acid Volume (mL)	Shaking frequency (rpm)	Testing instrument
071	EN 71-3:2013+A1:2014	--	0.30	15	500	ICP-OES/ ICP-MS
072	EN 71-3:2013+A1:2014	--	0.1999	7mL HNO ₃ (conc. & analytical)	--	ICP-OES
073	ISO 8124-3:2010+A1:2014	50	1	50	180	FAAS
074	ISO 8124-3:2010+A1:2014	50	0.1-0.107	5	6 degree	FAAS/ ICP-OES
075	ISO 8124-3:2010+A1:2014	100	1.0031	50	100	ICP-OES
076	ISO 8124-3:2010	100	0.3158	5	not monitored	ICP-OES
077	ISO 8124-3:2010	250	2	100	115 minutes with stirring rods	ICP-OES
078	EN 71-3:2013	30	0.25	12.5	80	ICP-MS
079	ASTM F963-11	70	0.4781	23.9608	120	ICP-OES
080	--	--	--	--	--	--
081	ISO 8124-3:2010+A1:2014	25	0.2000	10.0	110	ICP-OES
082	ISO 8124-3:2010+A1:2014	50	0.2	10	160	ICP-OES
083	ISO 8124-3:2010+A1:2014	5	0.1	5	120	ICP-OES
084	ISO 8124-3:2010+A1:2014	50	0.2004/ 0.2002	10/10	96	ICP-OES
085	ISO 8124-3:2010+A1:2014	100	0.50	25	120	ICP-OES
086	ISO 8124-3:2010+A1:2014	25	0.100	5.0	140	ICP-OES

Note: "--" Results or information were not provided.

Figure A.1 Z-scores of Migration of Barium

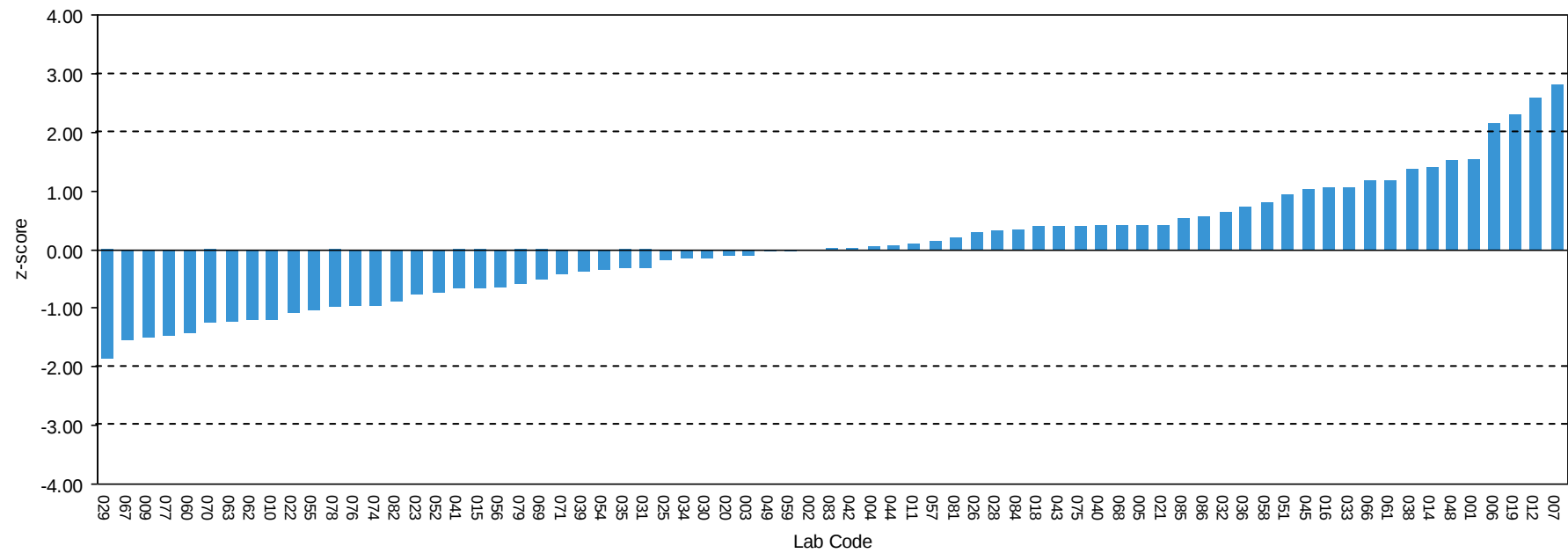


Figure A.2 Z-scores of Migration of Cadmium

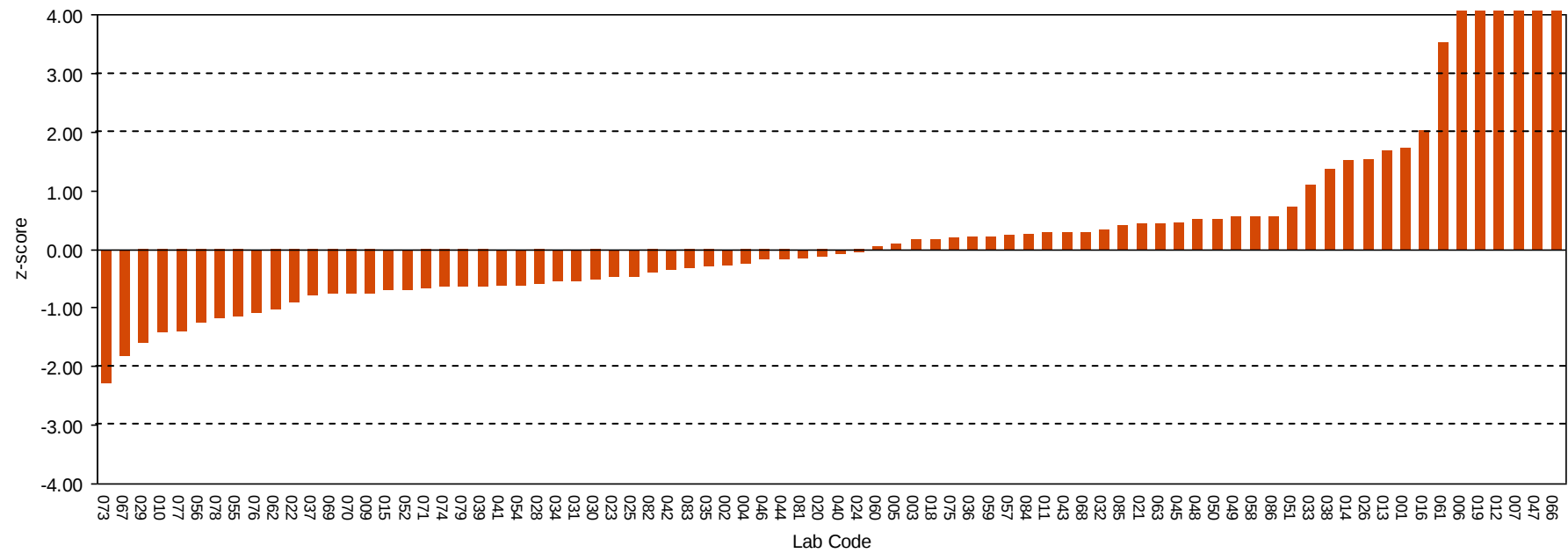


Figure A.3 Z-scores of Migration of Chromium

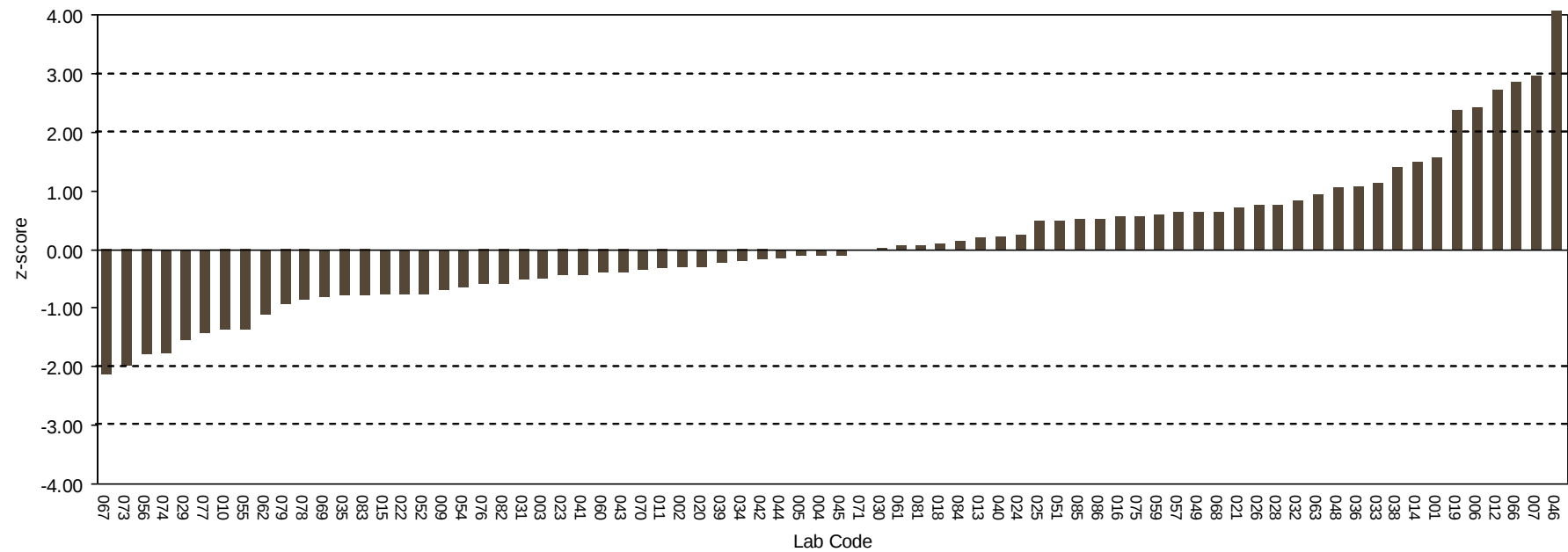


Figure A.4 Z-scores of Migration of Lead

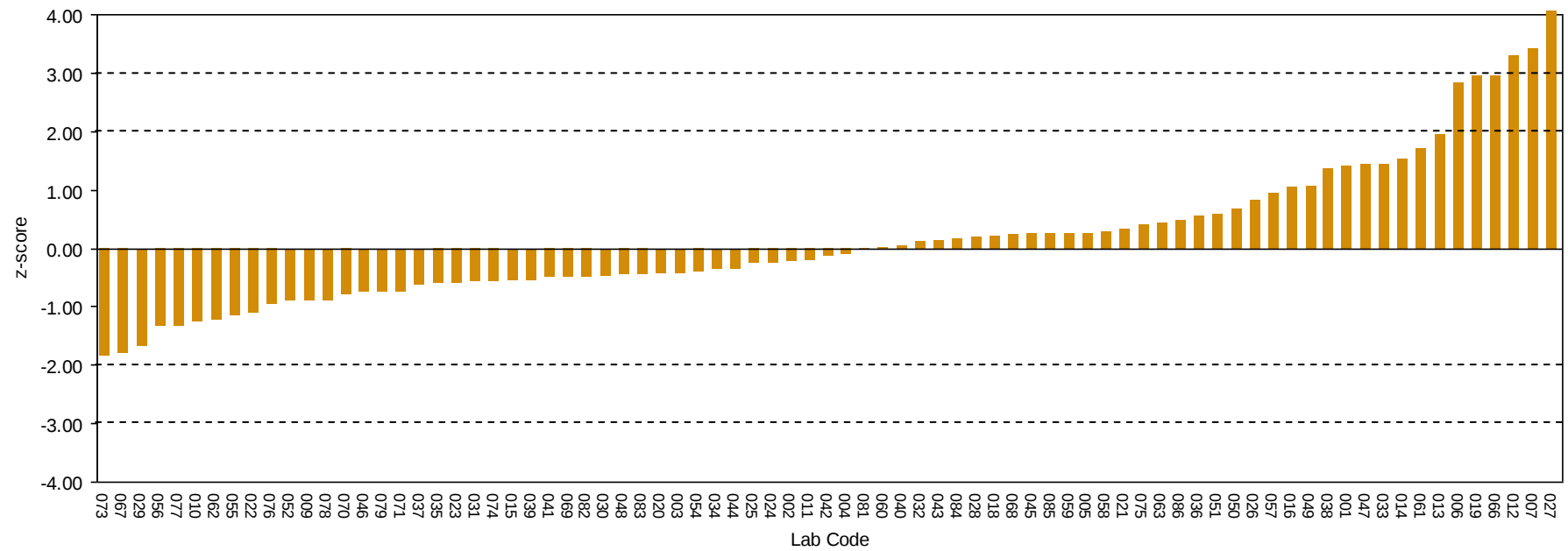
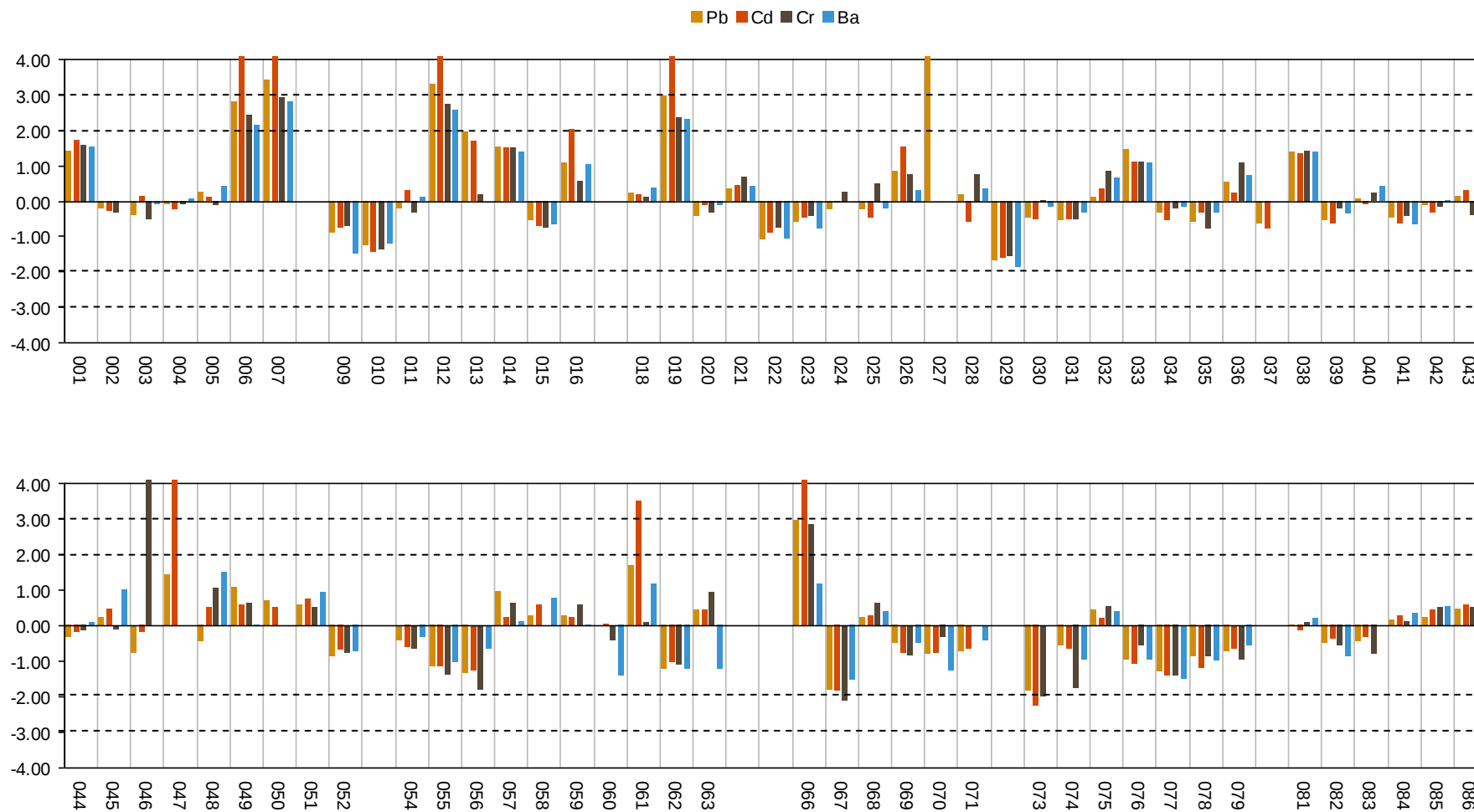


Figure A.5 Bar-chart of z-scores of a proficiency testing scheme in which the participants determined the concentrations of four elements



APPENDIX B Sample preparation and homogeneity study

1. Sample preparation

Weight certain heavy metal powder in container and place the container in oven at $(105 \pm 5)^\circ \text{C}$ for (30 ± 5) min. Remove container from the oven and allow to cool in a desiccators for (30 ± 5) min. After cooling, grinding and mixing the heavy metal powder uniformly in the mortar, and add it to the paint in high speed dispersion machine dispersed about 20 minutes, then add ball, scattered six hours, filter out the ball, the light oil, pigments, additives and color and yellow oil, mixed in high speed dispersion machine 12800 turn / min, dispersed 1 hour. With three roller machine after 3 times to reach the 7HA granularity, the grinding good paint to add thinner dispersed into a homogeneous liquid paint, the liquid paint in 140 degree oven baking 30 minutes, remove and cooling. Grinding and mixing the dry paint until homogenization, and then loading 2 grams / bottle.

2. Homogeneity test

2.1 Homogeneity check

10 proficiency test samples are selected by using a random selection method. 2 test portions are extracted from each bottle and tested in a random order, under repeatability conditions in accordance with the corresponding provisions for determination of migration of heavy metals in toys coating content in ISO 8124-3:2010+A1:2014. Determine the content of migration elements of Barium, Cadmium, Chromium and Lead by ICP-OES. The data are given in the Table below.

The procedure in Annex B of ISO 13528:2015 is followed, resulting in the summary statistics listed. The fitness-for purpose σ_{pt} of Ba, Cd, Cr, Pb is the robust standard deviation of participant results, so the estimate of sample variability is checked against 0.3 times the σ_{pt} .

Table B.1 Homogeneity data for proficiency test items

Bottle ID	Result of Ba (mg/kg)		Result of Cd (mg/kg)	
1	298.9	292.2	20.5	19.8
2	299.4	293.2	20.4	19.9
3	292.2	301.5	20.1	20.6
4	301.2	297.2	20.7	20.4
5	301.3	301.2	20.5	20.7
6	301.0	298.2	20.6	20.1
7	302.2	306.1	20.4	20.7
8	294.3	298.6	20.0	20.5
9	295.5	290.7	19.4	19.5
10	296.9	300.2	20.2	20.0

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Bottle ID	Result of Cr (mg/kg)		Result of Pb (mg/kg)	
1	25.5	24.3	74.2	71.8
2	25.7	24.2	75.9	72.7
3	24.8	25.7	73.6	75.7
4	25.0	24.7	75.7	74.0
5	25.4	24.8	75.3	74.6
6	25.4	25.7	76.3	74.4
7	25.8	25.8	76.0	77.4
8	24.4	25.1	73.0	75.3
9	24.2	24.1	74.0	70.8
10	24.9	26.6	74.9	75.7

2.2 Results

Table B.2 Homogeneity results for proficiency test items

Measurands	Result (mg/kg)			
	Ba	Cd	Cr	Pb
Overall average (X)	298.1	20.3	25.1	74.6
SD	4.03	0.39	0.68	1.61
s_x	3.14	0.33	0.51	1.22
s_w	3.61	0.30	0.64	1.50
s_s	1.83	0.25	0.23	0.60
σ_{pt}	82.6	6.10	6.16	24.0
$0.3^* \sigma_{pt}$	24.8	1.83	1.85	7.20

2.3. Evaluation of homogeneity

s_s of Ba, Cd, Cr, Pb is less than the check values($0.3^* \sigma_{pt}$), so homogeneity is sufficient. Therefore it was concluded that any outlier results subsequently identified could not be attributed to sample variability.

3. Stability test

3.1 Stability check

There is reasonable assurance from previous experience that instability is unlikely. The procedure in Annex B of ISO 13528:2015 is followed to evaluate stability. 3 proficiency test items are randomly selected and stored at room temperature. The proficiency test items were tested in duplicate, and the six results are checked against the homogeneity values.

Table B.3 Stability data for proficiency test items

Bottle ID	Result of Ba (mg/kg)		Result of Cd (mg/kg)	
1	305.6	311.5	19.8	19.9
2	307.1	303.0	19.9	19.6
3	306.1	312.7	19.7	20.2
Bottle ID	Result of Cr (mg/kg)		Result of Pb (mg/kg)	
1	26.4	27.4	72.7	74.0
2	26.6	26.3	73.3	72.4
3	26.4	27.3	74.6	74.7

3.2 Results

Table B.4 Stability results for proficiency test items

Measurands	Result (mg/kg)			
	Ba	Cd	Cr	Pb
Average of Homogeneity	298.1	20.3	25.1	74.6
Average of Stability	307.7	19.9	26.7	73.6
Absolute value of difference	9.6	0.4	1.6	1.0
Check value($0.3 * \sigma_{pt}$)	24.8	1.8	1.8	7.2

3.3 Evaluation of stability

Compare the general average of the measurements obtained in the homogeneity check with the general average of the results obtained in the stability check. As the absolute value of difference between average of homogeneity and average of stability is less than the check value($0.3 * \sigma_{pt}$), so the samples are considered to be adequately stable.



APPENDIX C Relevant documents

Sample Receipt Form (Participating Laboratories)

Institute/
Laboratory: _____

Postal
address: _____

Lab Code: _____

Contact
person: _____

Title	Given name	Surname
_____	_____	_____

E-mail: _____

Print name /
Signature: _____

Date: _____

Confirmation of Package Contents

I hereby acknowledge the receipt of package of APLAC proficiency testing programme (APLAC T098).

☐ Do you receive the electronical copy of result proforma and instruction from AB? ☐ Yes ☐ No

☐ The sample is *Intact & Sealed / Broken / Missing** and should be *Suitable / Not Suitable** for analysis (* Please delete as appropriate).

Other
comments: _____

Upon receipt of the sample, please complete this form and return it to the co-ordinator of the proficiency testing programme (E-mail: liuch@iqtc.cn heping@cnas.org.cn).



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**Sample Receipt Form
(Accreditation Bodies)**

Accreditation
Body:

Postal address:

Contact person:

Title

Given name

Surname

E-mail:

Print name /

Signature:

Date:

Confirmation of Package Contents

I hereby acknowledge the receipt of the sealed package containing the proficiency test sample(s) for the APLAC proficiency testing programme (APLAC T098).

The sample(s) is/are *Intact & Sealed* / *Broken* / *Missing** and should be *Suitable* / *Not Suitable** for analysis. (* Please delete as appropriate)

Other comments:

Upon receipt of the sample(s), please complete this form and return it to the co-ordinator of the proficiency testing programme (E-mail: liuch@iqtc.cn heping@cnas.org.cn).



Instruction for Accreditation Bodies

1. Distribution of the proficiency test sample

Accreditation bodies (AB) should receive the sealed package containing the proficiency test samples and the instructions (softcopy) from the organizers. Upon receipt of the samples, please complete the Sample Receipt Form (Accreditation Bodies) and return it to the coordinators of the proficiency testing programme. Replacement will be arranged if the proficiency test samples are identified to be not suitable for analysis.

AB should distribute the proficiency test samples to the nominated laboratories as soon as possible and inform your nominated laboratory about the **code number** individually.

AB should request the nominated laboratories to carefully check the proficiency test samples upon receipt. Also, AB should ask the nominated laboratories to complete the Sample Receipt Form (Participating Laboratories) and return it to the coordinators of the proficiency testing programme

2. Reporting and submission of results

AB shall remind the nominated laboratories to complete the *Result Proforma* and submit it electronically to the coordinators of the proficiency testing programme on or before the deadline **June 30th 2015**. Under normal circumstances, results submitted after the deadline will NOT be accepted.

3. Contact

AB may wish to contact the coordinators of the proficiency testing programme for any enquires (E-mail: liuch@iqtc.cn heping@cnas.org.cn).



Instruction for Participating Laboratories
--

Dear participant:

Welcome to **Proficiency Testing Program APLAC T098 Determination of Migration of Heavy Metals in Toys Coating.**

To ensure smooth implementation of this program, please read the following instructions carefully before testing.

For the sake of confidentiality, each participant is granted a code, which will be used in the evaluation. Participants will receive the code number of their own from AB. In case you have any questions, please contact us.

1. Analysis of the proficiency test sample

Participating laboratories will be provided with ONE brown bottle containing about approx. 2 grams of toy painting powder numbered **H1504A**. The PT sample has already been crushed and passed through 500 µm sieve, so there is no need to reprepare the sample. Upon receipt of the sample, please complete the Sample Receipt Form (Participating Laboratories) and return it to the co-ordinator of the proficiency testing programme. Replacement of a new bottle of sample will be arranged if the proficiency test sample is identified to be not suitable for analysis.

Please prepare the sample according to the relevant clauses in **ISO 8124-3:2010+A1:2014** or other appropriate method, and determine the migration content of the elements Barium, Cadmium, Chromium and Lead by appropriate instrument. Please treat the sample as if it is a routine sample and analyze it in the way you would normally do in day-to-day circumstances. Please determine the sample twice in parallel and submit the result in Result Proforma. For this proficiency testing programme, return of the proficiency test sample is not necessary.

2. Reporting and submission of results

Participants should Report your analytical results to one decimal place in the “Result Proforma” (for example: XXX.X or XX.X). Please do not report ‘less than’-results which are above your detection limit. Results reported as ‘less than’ cannot be used for meaningful statistical calculations.

Participants should be aware that any submitted results are considered final and accordingly such results and units should be thoroughly checked before submission. Participants should submit the Result Proforma electronically to the co-ordinator of the proficiency testing programme on or before the deadline **June 30th 2015**. Results submitted after the deadline will not be accepted. Participants are reminded



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that the ability to report results in the specified unit and within the given time scale are part of the proficiency test.

An electronic copy of the final report on the performance of participating laboratories would be distributed to the participants in December 2015. The report will reveal only the code number assigned to the designated participating laboratory and the identity of participants in this proficiency testing programme will be kept confidential.

The proficiency testing programme is conducted in the belief that participants will perform the analysis and report results with scientific rigour. Collusion and falsification of results are clearly against the spirit of the proficiency testing programme.

3. Contact

Participants may wish to contact the coordinators of the proficiency testing programme for any enquires.

The contact details are given below:

Coordinator of organising accreditation body CNAS:

Name: HE Ping (Mr.)

Add.: No.8 Nanhuashi, Dongcheng District, Beijing, 100062, China

E-mail: heping@cnas.org.cn

Tel: +8610-67105290

Coordinator of the proficiency testing provider in IQTC:

Name: Liu Chonghua

Add.: 17/F, B Tower, No. 66 Huachang Avenue, Zhujiang New Town, Guangzhou, China

E-mail: liuch@iqtc.cn

Tel: +8620-38290540



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Result Proforma

■ Information of participant:

Lab Code:		Country:		City:	
Name of laboratory:					
Contact Address:					
Contact Person:		Postcode:			
Contact Phone:		Fax:			
Signature of responsible manager:		Date of report:			

■ Result Report on H1504A pink toys coating (unit:mg/kg):

Element	Migration of Barium	Migration of Cadmium	Migration of Chromium	Migration of Lead
Result 1 *				
Result 2 *				
Average Result				
Expanded Measurement Uncertainty				
MDL				

Remarks:

- * Report result values to one decimal place.(for example: XXX.X or XX.X)
- Please email the completed report form to the co-ordinator of the proficiency testing programme (E-mail: liuch@iqtc.cn heping@cnas.org.cn) **before June 30th, 2015.**

■ **Test Parameters:**

Please tick the appropriate option, and specify the details with words if necessary.

1. Which testing standard method(s) was(were) used to determine the elements?

- ☐ ISO 8124-3:2010+A1:2014
- ☐ GB 6675-2014
- ☐ EN 71-3:2013+A1:2014
- ☐ ASTM F963-11
- ☐ Others _____ (Please specify)

2. Specify the extraction condition:

- ☐ Name of extraction vessel: _____ Volume: _____ mL

3. After extraction, has the solution been diluted before determination?

- ☐ No.
- ☐ Yes. Dilution ratio: _____ Matrix of solvent used for dilution: _____

4. Specify the conditions of sample extraction:

- ☐ Weight of test portion _____ g Volume of acid _____ mL

5. Specify the concentration of hydrochloric acid used for extraction:

- ☐ $c(\text{HCl}) =$ _____ mol/L (3 significant figures)



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6. Has the acidity been adjusted?

- ☐ No. The pH value directly found to be_____
- ☐ Yes. The pH was adjusted to_____

7. Which means was used to agitate the mixture?

- ☐ Constant temperature shaking water bath, shaking frequency was_____rotations per minute(rpm)
- ☐ Operated by manual, shaking frequency was_____rotations per minute(rpm)
- ☐ Other device, _____ (please specify), shaking frequency was_____rotations per minute(rpm)

8. Which testing instrument was used to determine the migration elements? Please also specify the instrument type and working condition.

☐ FAAS : ☐ Ba ☐ Cd ☐ Cr ☐ Pb Instrument type_____

Working condition_____(Please specify)

☐ ICP-OES : ☐ Ba ☐ Cd ☐ Cr ☐ Pb Instrument type_____

Working condition_____(Please specify)

☐ ICP-MS : ☐ Ba ☐ Cd ☐ Cr ☐ Pb Instrument type_____

Working condition_____(Please specify)

☐ Others : ☐ Ba ☐ Cd ☐ Cr ☐ Pb Instrument type_____

Working condition_____(Please specify)



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9. Did your testing processes have any deviation from the specified details of standard method? If you have, please indicate as clearly as possible.

☐ No.

☐ Yes.

(Please specify)

10. Please specify your suggestions or comments if you have.

(Please specify)

End of Report