Asia Pacific Laboratory Accreditation Cooperation

APLAC T051 Heavy Metal Elements in Plastic Proficiency Testing Program

May 2007

ACKNOWLEDGEMENTS

The APLAC secretariat wishes to acknowledge gratefully Plastics Industry Development Center, R.O.C. for coordinating the supply, packaging and pre-testing of the samples and technical advice for this program. Thank you to Mr. Ming-Kun, Chen（PIDC） for acting as a technical adviser for this program.

CONTENTS

1 FOREWORD

This report summarises the results of APLAC T051 of Heavy Metal Elements in Plastics Proficiency Testing Program involving testing laboratories for the Asia Pacific Laboratory Accreditation Cooperation (APLAC) and European co-operation for Accreditation (EA) and InterAmerican Accreditation Cooperation (IAAC) and unaffiliated ILAC bodies. This program covered analysis chemical compositions in plastics included Lead, Cadmium, Chromium and Mercury contents.

The program was conducted by the CNLA/ TAF (Taiwan Accreditation Foundation). The aim of the program was to assess laboratories' ability to competently perform the tests examined.

2 FEATURES OF THE PROGRAM

(a) APLAC and EA members were invited to participate. There were in total 33 laboratory participants nominated by 15 accreditation bodies (ABs) in this program. All samples were sent to the ABs and transferred to the participating laboratories. Two participating laboratories did not return results; hence the results from those laboratories were excluded from the analysis.

Economy	No. of Labs	Economy	No. of Labs
Bulgaria	1	Mexico	1
Chile	3	Poland	2
Czech	4	Portugal	2
Germany	1	Singapore	3
India	1	Slovakia	2
Indonesia	2	Taiwan	4
Israel	2	Thailand	3
Japan	2	Total	33

(b) Participating laboratories were supplied two sealed amber bottles containing about 7 g plastic particle sample with labelled APLAC T051 A# and APLAC T051 B# respectively.

(c) Each participating laboratory is required to determine the level (ppm or mg/kg) of Lead content, Cadmium content, Chromium content and Mercury content in sample A and sample B.

(d) Prior to distribution the samples were analysed for homogeneity. The testing results showed that the samples were sufficiently homogeneous. Therefore any results later identified as outliers could not be attributed to any significant sample variability. (Appendix B)

(e) Laboratories were requested to perform the tests according to their copy of the "Instructions to Participants" and to record their results on the accompanying "Results Sheet", both of which were distributed to participants with the sample. (Appendix C)

(f) Each laboratory was randomly allocated a unique code number for the program to enable confidentiality of results. Its code number makes reference to each laboratory in this report.

3 CONTENT OF APPENDICIES

APPENDIX A contains:

(a) The duplicate results reported by laboratories and the calculated Z-Score result for each test on each sample.

(b) A table of robust statistics for each test - number of results, median, normalised IQR, robust CV, minimum, maximum and range.

(c) Z-Scores and ordered Z-Score charts calculated for laboratories for each test.

(d) Test methods are reported for each lab.

APPENDIX B contains details of sample preparation and homogeneity testing.

APPENDIX C contains a copy of the "Instructions to Participants" and the "Results Sheet", as supplied to participants.

APPENDIX D contains details of statistical procedures, calculations and formulae.

4 STATISTICAL DESIGN OF THE PROGRAM

A split-level statistical design was used in this program. The samples labelled APT51A, and APT51B were different concentrations.

Robust statistical procedures were used to generate the Z-Scores and summary statistics for the sample - number of results, median, normalised interquartile range, minimum, maximum and range.

For each laboratory, a single robust Z-Score was calculated for each sample.

TABLE A: SUMMARY STATISTICS

Test	Sample	Summary Statistics		
		No of Results	Median	Normalised IQR
Lead Content	A	25	95.9	16.46
	B	24	14.55	2.91
Cadmium Content	A	31	129.5	17.20
	B	31	20.5	2.00
Chromium Content	A	26	107.65	10.36
	B	26	17.3	2.30
Mercury Content	A	22	24.6	2.97
	B	21	4.4	0.45

5 OUTLIER RESULTS

In order to achieve the program's aim of assessing laboratories' testing performance, a robust statistical approach, which uses Z-Scores to assess participants performance has been utilised. The Z-Score is a measure of how far the result(s) is from the consensus value - a normalized value that gives a "score" to each result relative to the other results in the group. Therefore a Z-Score close to zero means that the result agrees well with those from other laboratories. An outlier will be any result(s) that has an absolute Z-Score value greater than three.

According to the definition of ISO/IEC Guide 43-1, extreme results are outliers and other values that are grossly inconsistent with other members of the data set. In other words, extreme results may be outliers, which are erroneously reporting the presence/absence of an analyst, or other gross errors which can not be included in the statistical analysis, for example: ND (denotes no detect), “<15” or “<0.5”.

Appendix D gives details on the calculation of Z-Scores.

TABLE B: Laboratory’s Code of Outliers

Test	Sample	Lab Code
Lead Content	A	4,27,28
	B	4,6,20
Cadmium Content	A	4,6,27,33
	B	4,6,20,22,27
Chromium Content	A	4, 28
	B	4, 27
Mercury Content	A	1, 22,26
	B	1, 20,22,25,26,28

Of the 31 participating laboratories, the total of 10 laboratories has been identified as having reported one or more outlier results. Out of the 206 results returned, the total of 28 results has been identified as outlier results (13.59 %).

6 TECHNICAL COMMENTS

General Comments

According to terms of use and as indicated by statistical data results, outliers' pre-treatment or conditions were different from common methods in force. Moreover, equipments adopted (GFAA, ICP-OES or XRF) in analysis were also different. All these had influenced the final results to certain degree. Although the pre-treatment and equipments could cause influence, the results and theories showed/ corroborated that they are as the same as the integral results.

(a) Pb

Lab code No. 4, which tested element Pb and were classified outliers in both high and low concentration, as an example. We suggest that the sample pre-treatment, testing processes, data analysis and data processing should be checked carefully. As for labs that were classified outliers in low concentration but satisfied in high one, i.e. No. 6 and No. 20, it is suggested to lift the sensitivity and detection limits of Pb.

(b) Cd

Labs code No.4, 6 and 27 should check the integral process of the test in order to find out the reasons that caused outliers.

(c) Cr

Total Cr was used for the analysis in this program. Lab code No. 25 specified Cr^{6+} as the element they tested, however, the data showed ND (Not Detected). Lab code No. 4 seems that reversed the data of Sample A and Sample B.

(d) Hg

The pre-treatment and test temperature would probably have great influence on the concentration of Hg. For instance, outlier laboratories adopted different pre-treatments from the qualified ones, as disclosed by the statistical results. Labs code No. 1,22 and 26 had very low results for both samples, but there were also several high outliers for the second sample.

APPENDIX A

Summary of Results

Test Methods

A1.1 Pb (A)

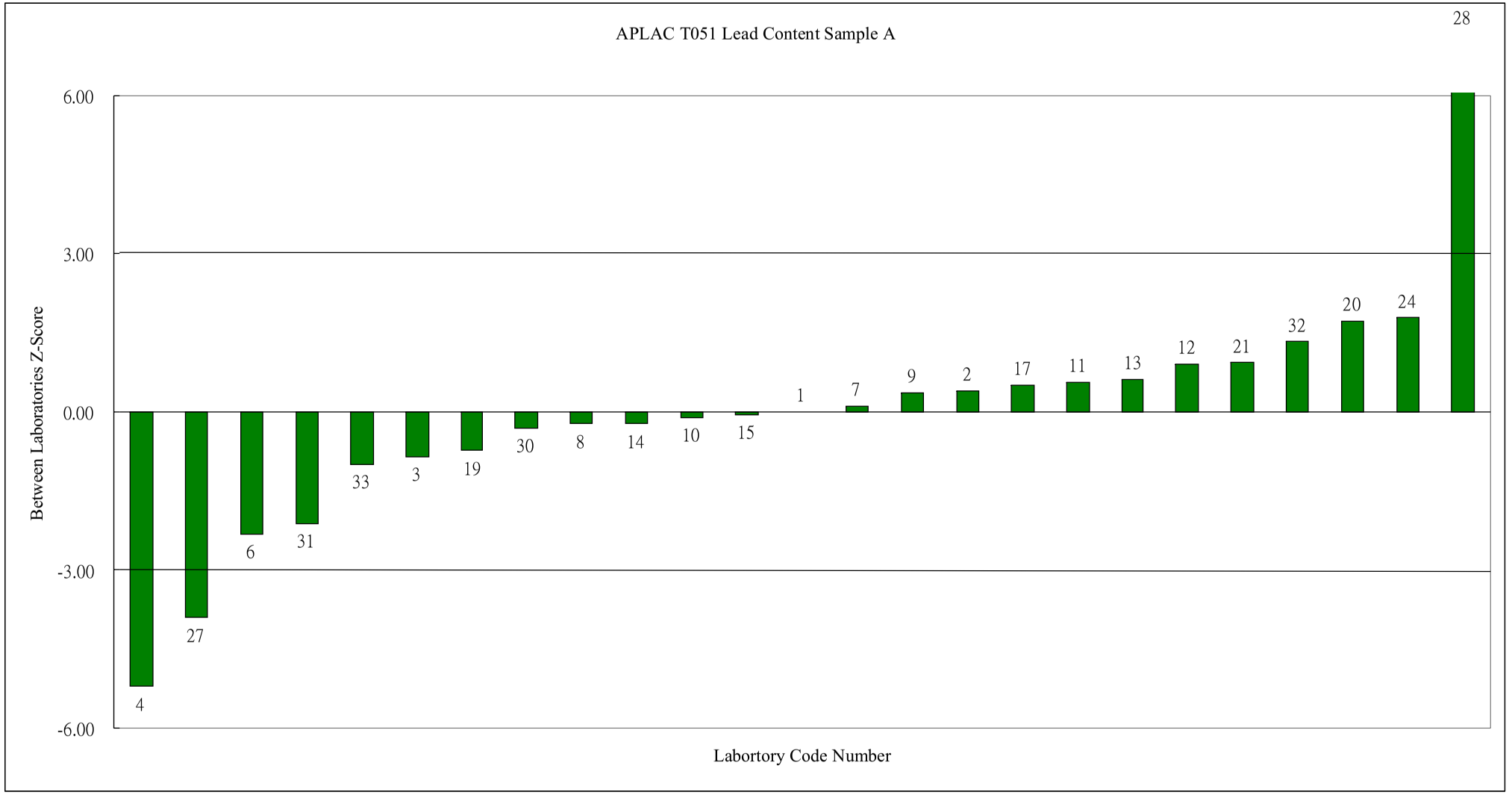
Lab Code	Result (ppm)	Z-Score	Expanded Uncertainty
1	95.9	0.00	±2.4100
2	102.3	0.39	0.068
3	82.1	-0.84	N/A
4	10.1	-5.21※	±2.3
5	N/A	N/A	N/A
6	57.6	-2.33	N/A
7	97.9	0.12	±18.7%
8	92	-0.24	N/A
9	101.8	0.36	±0.4
10	93.9	-0.12	±0.176
11	105	0.55	7.5
12	110.7	0.90	N/A
13	106.1	0.62	1.52
14	92.4	-0.21	0.0406
15	95.1	-0.05	6.6
16	N/A	N/A	N/A
17	104.3	0.51	14.0
18	<1.5	N/A	N/A
19	83.9	-0.73	25%
20	124.4	1.73	±40.7
21	111.4	0.94	1.7(%)
22	<0.01	N/A	N/A
23	N/A	N/A	N/A
24	125.2	1.78	17.6%
25	ND	N/A	±6.6
26	<0.5	N/A	N/A
27	31.6	-3.91※	2.1
28	258.8	9.90※	17%
29	<15	N/A	±10%
30	90.7	-0.32	26.6%
31	61	-2.12	4.8
32	117.6	1.32	14.1
33	79.7	-0.98	4.0

No. of result	25
Median	95.9
Normalized IQR	16.46
Robust CV (%)	17.2
Maximum	258.8
Minimum	10.1
Range	248.7

NOTE:

1. ※ Denotes an outlier (i.e. | Z-Score| ≧ 3)
2. ND denotes not detect

A1.2


APLAC T051 Lead Content Sample A
Between Laboratories Z-Score
6.00
3.00
0.00
-3.00
-6.00
4
27
6
31
33
3
19
30
8
14
10
15
1
7
9
2
17
11
13
12
21
32
20
24
28
Labortory Code Number

2.1 Pb (B)

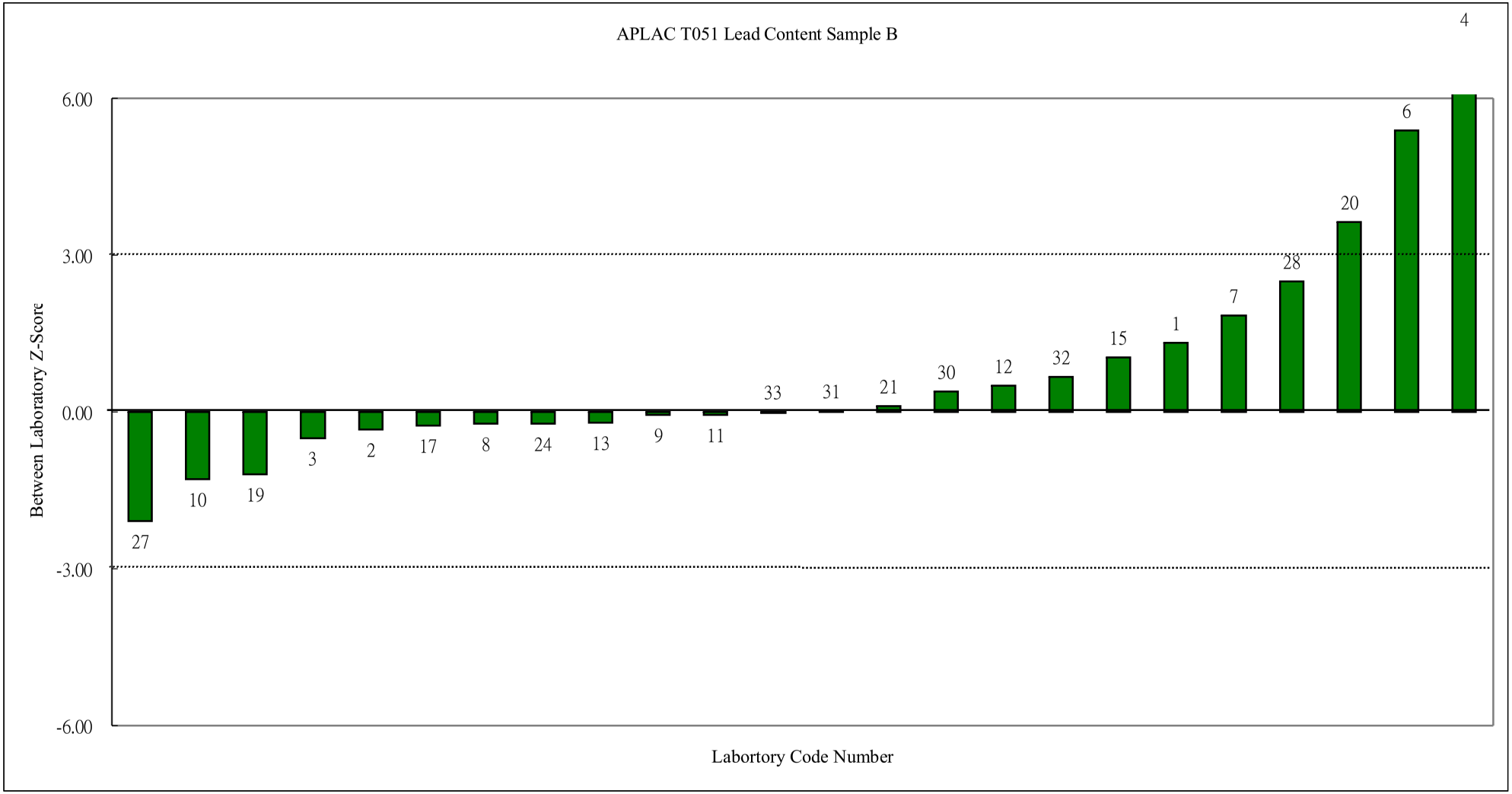
Lab Code	Result (ppm)	Z-Score	Expanded Uncertainty
1	18.4	1.32	±0.4663
2	13.6	-0.33	0.068
3	13.1	-0.50	N/A
4	35.3	7.13※	±2.3
5	N/A	N/A	N/A
6	30.2	5.38※	N/A
7	19.9	1.84	±18.7%
8	13.9	-0.22	N/A
9	14.4	-0.05	±0.4
10	10.8	-1.29	±0.11
11	14.4	-0.05	7.5
12	16	0.50	N/A
13	13.96	-0.20	1.52
14	ND	N/A	0.0406
15	17.6	1.05	3.0
16	N/A	N/A	N/A
17	13.8	-0.26	1.2
18	<1.5	N/A	N/A
19	11.1	-1.19	25%
20	25.1	3.63※	±17.6
21	14.9	0.12	2.4%
22	<0.01	N/A	N/A
23	N/A	N/A	N/A
24	13.9	-0.22	17.6%
25	ND	N/A	±6.6
26	<0.5	N/A	N/A
27	8.5	-2.08	0.6
28	21.8	2.49	17%
29	<15	N/A	±10%
30	15.7	0.40	26.6%
31	14.6	0.02	4.6
32	16.5	0.67	2.5
33	14.5	-0.02	1.16

No. of result	24
Median	14.55
Normalized IQR	2.91
Robust CV (%)	20.0
Maximum	35.3
Minimum	8.5
Range	26.8

NOTE:

1. ※ Denotes an outlier (i.e. | Z-Score| ≧ 3)
2. ND denotes not detect

A2.2


APLAC T051 Lead Content Sample B
6.00
3.00
0.00
-3.00
-6.00
Between Laboratory Z-Score
Labortory Code Number
27
10
19
3
2
17
8
24
13
9
11
33
31
21
30
12
32
15
1
7
28
20
6
4

A3.1 Cd (A)

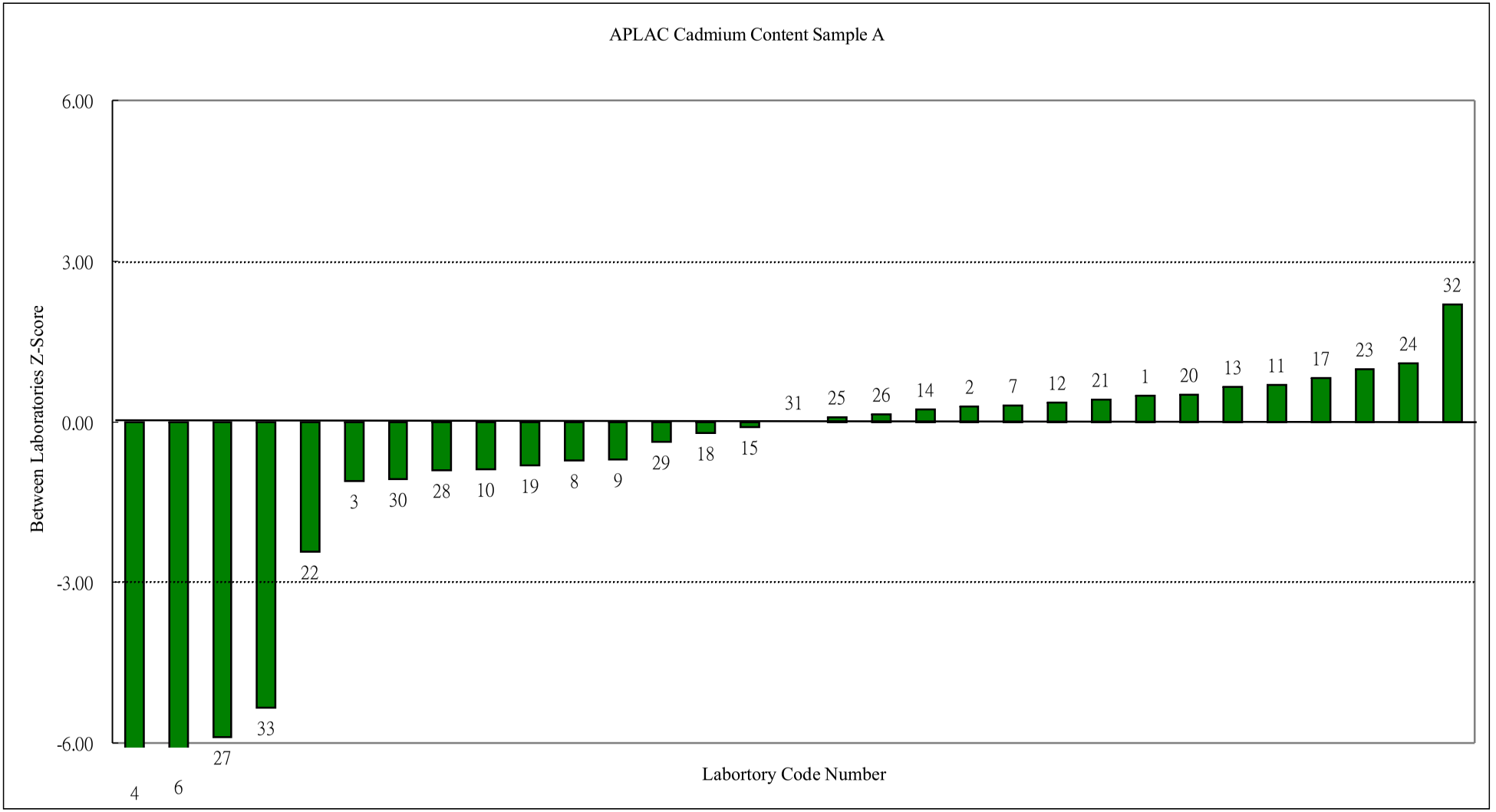
Lab Code	Result (ppm)	Z-Score	Expanded uncertainty
1	137.8	0.48	±0.9538
2	134.5	0.29	0.028
3	110.5	-1.10	N/A
4	5.8	-7.19※	±1.7
5	N/A	N/A	N/A
6	18.6	-6.45※	N/A
7	134.6	0.30	±16.6%
8	117.1	-0.72	N/A
9	117.3	-0.71	±0.4
10	114.1	-0.90	±2.6
11	141.2	0.68	4.9
12	135.8	0.37	N/A
13	140.7	0.65	1.12
14	133.3	0.22	0.0406
15	127.8	-0.10	7.5
16	N/A	N/A	N/A
17	143.6	0.82	10.9
18	126	-0.20	15(±U v%)
19	115.5	-0.81	25%
20	138.1	0.50	±16.1
21	136.5	0.41	1.8%
22	87.9	-2.42	9.3
23	146.3	0.98	13.5
24	148.3	1.09	14.6%
25	130.9	0.08	±8.5
26	131.8	0.13	±33
27	28.1	-5.90※	2.9
28	113.8	-0.91	17%
29	122.9	-0.38	±10%
30	111	-1.08	30.0%
31	129.5	0.00	3.4
32	167.2	2.19	25.1
33	37.8	-5.33※	2.27

No. of result	31
Median	129.5
Normalized IQR	17.20
Robust CV (%)	13.3
Maximum	167.2
Minimum	5.8
Range	161.4

NOTE:

1. ※ Denotes an outlier (i.e. | Z-Score| ≧ 3)
2. ND denotes not detect

A3.2


APLAC Cadmium Content Sample A
Between Laboratories Z-Score
6.00
3.00
0.00
-3.00
-6.00
4
6
27
33
22
3
30
28
10
19
8
9
29
18
15
31
25
26
14
2
7
12
21
1
20
13
11
17
23
24
32
Labortory Code Number

A4.1 Cd (B)

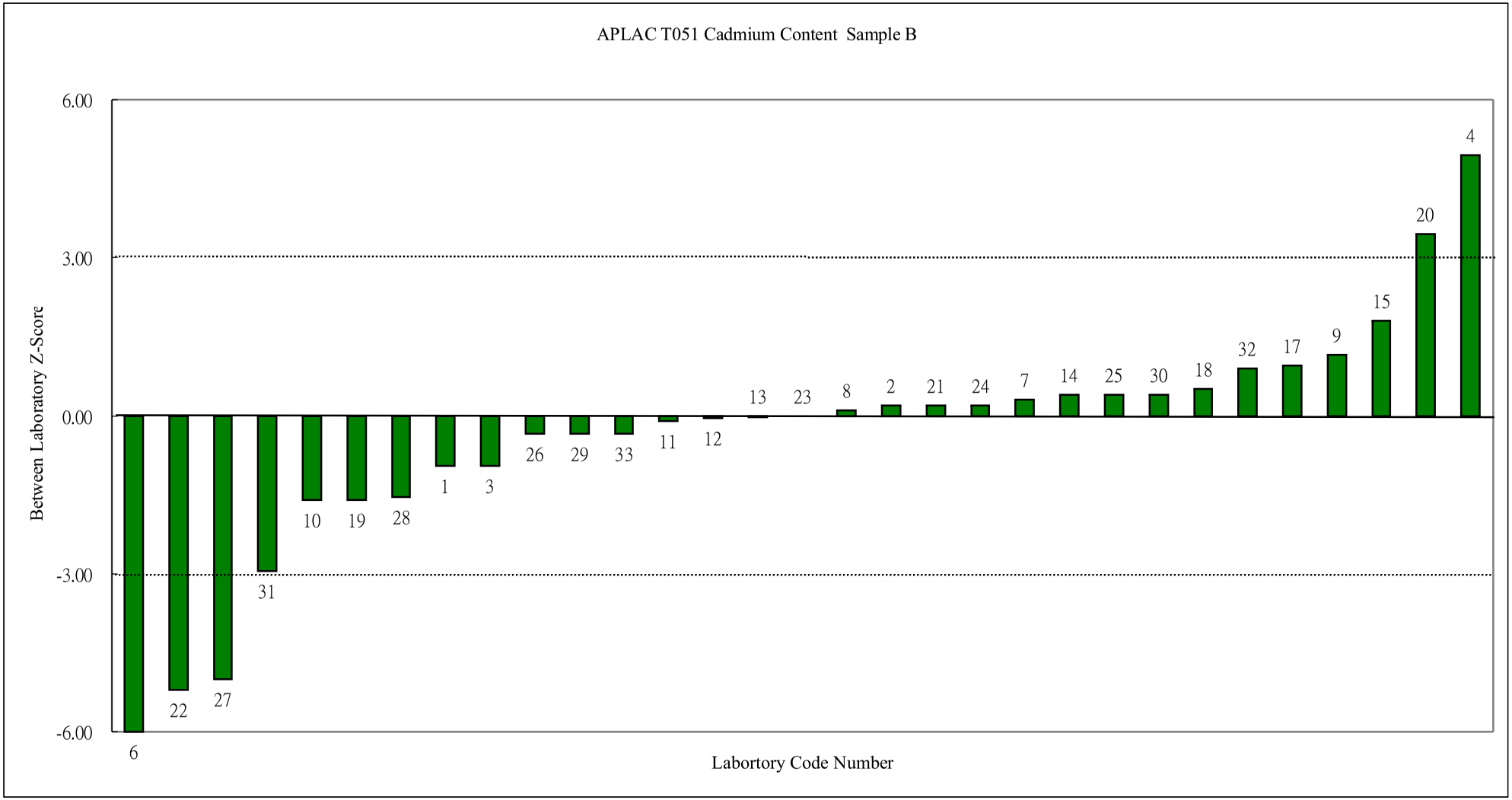
Lab Code	Result (ppm)	Z-Score	Expanded Uncertainty
1	18.6	-0.95	±0.4033
2	20.9	0.20	0.028
3	18.6	-0.95	N/A
4	30.4	4.95※	±1.7
5	N/A	N/A	N/A
6	8.5	-6.00※	N/A
7	21.1	0.30	±16.6%
8	20.7	0.10	N/A
9	22.8	1.15	±0.4
10	17.3	-1.60	±0.9
11	20.3	-0.10	4.9
12	20.4	-0.05	N/A
13	20.43	--0.03	1.12
14	21.3	0.40	0.0406
15	24.1	1.80	1.6
16	N/A	N/A	N/A
17	22.4	0.95	2.1
18	21.5	0.50	15 (±U v%)
19	17.3	-1.60	25%
20	27.4	3.45※	±12.2
21	20.9	0.20	2.2%
22	10.1	-5.20※	0.5
23	20.5	0.00	2.3
24	20.9	0.20	14.6%
25	21.3	0.40	±2.9
26	19.8	-0.35	±6.9
27	10.5	-5.00※	1.0
28	17.4	-1.55	17%
29	19.8	-0.35	±10%
30	21.3	0.40	30.0%
31	14.6	-2.95	2.4
32	22.3	0.90	3.3
33	19.8	-0.35	1.78

No. of result	31
Median	20.5
Normalized IQR	2.00
Robust CV (%)	9.8
Maximum	30.4
Minimum	8.5
Range	21.9

NOTE:

1. ※ Denotes an outlier (i.e. | Z-Score| ≧ 3)
2. ND denotes not detect

A4.2


APLAC T051 Cadmium Content Sample B
6.00
3.00
0.00
-3.00
-6.00
Between Laboratory Z-Score
6
22
27
31
10
19
28
1
3
26
29
33
11
12
13
23
8
2
21
24
7
14
25
30
18
32
17
9
15
20
4
Labortory Code Number

A5.1 Cr (A)

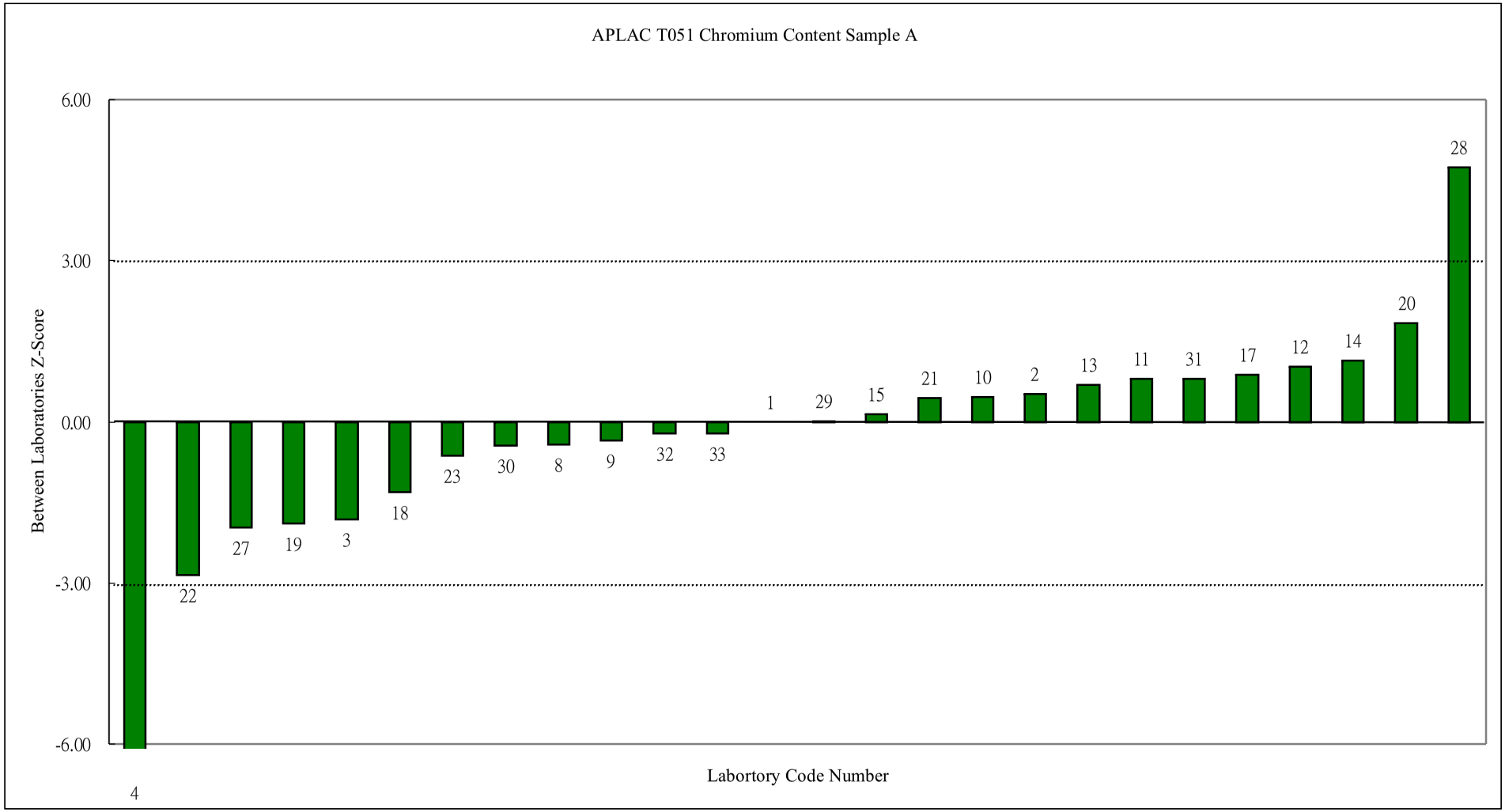
Lab Code	Result (ppm)	Z-Score	Expanded Uncertainty
1	107.5	-0.01	±0.8661
2	113	0.52	0.036
3	88.9	-1.81	N/A
4	22.3	-8.24※	±2.2
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	ND<2	N/A	N/A
8	103.2	-0.43	N/A
9	104.1	-0.34	±0.4
10	112.4	0.46	±2.15
11	115.9	0.80	4.1
12	118.2	1.02	N/A
13	114.8	0.69	1.90
14	119.4	1.13	0.0406
15	109.1	0.14	4.4
16	N/A	N/A	N/A
17	116.8	0.88	9.6
18	94.1	-1.31	30 (±U v%)
19	88.1	-1.89	25%
20	126.7	1.84	±36.9
21	112.2	0.44	4.2%
22	78.1	-2.85	10.5
23	101.2	-0.62	6.5
24	N/A	N/A	N/A
25	ND	N/A	N/A
26	<8	N/A	N/A
27	87.2	-1.97	11.4
28	156.7	4.73※	17%
29	107.8	0.01	±10%
30	103	-0.45	16.8%
31	115.9	0.80	3.4
32	105.5	-0.21	12.7
33	105.5	-0.21	7.4

No. of result	26
Median	107.65
Normalized IQR	10.36
Robust CV (%)	9.6
Maximum	156.7
Minimum	22.3
Range	134.4

NOTE:

1. ※ Denotes an outlier (i.e. | Z-Score| ≧ 3)
2. ND denotes not detect

A5.2


APLAC T051 Chromium Content Sample A
6.00
3.00
0.00
-3.00
-6.00
Between Laboratories Z-Score
4
22
27
19
3
18
23
30
8
9
32
33
1
29
15
21
10
2
13
11
31
17
12
14
20
28
Labortory Code Number

A6.1 Cr (B)

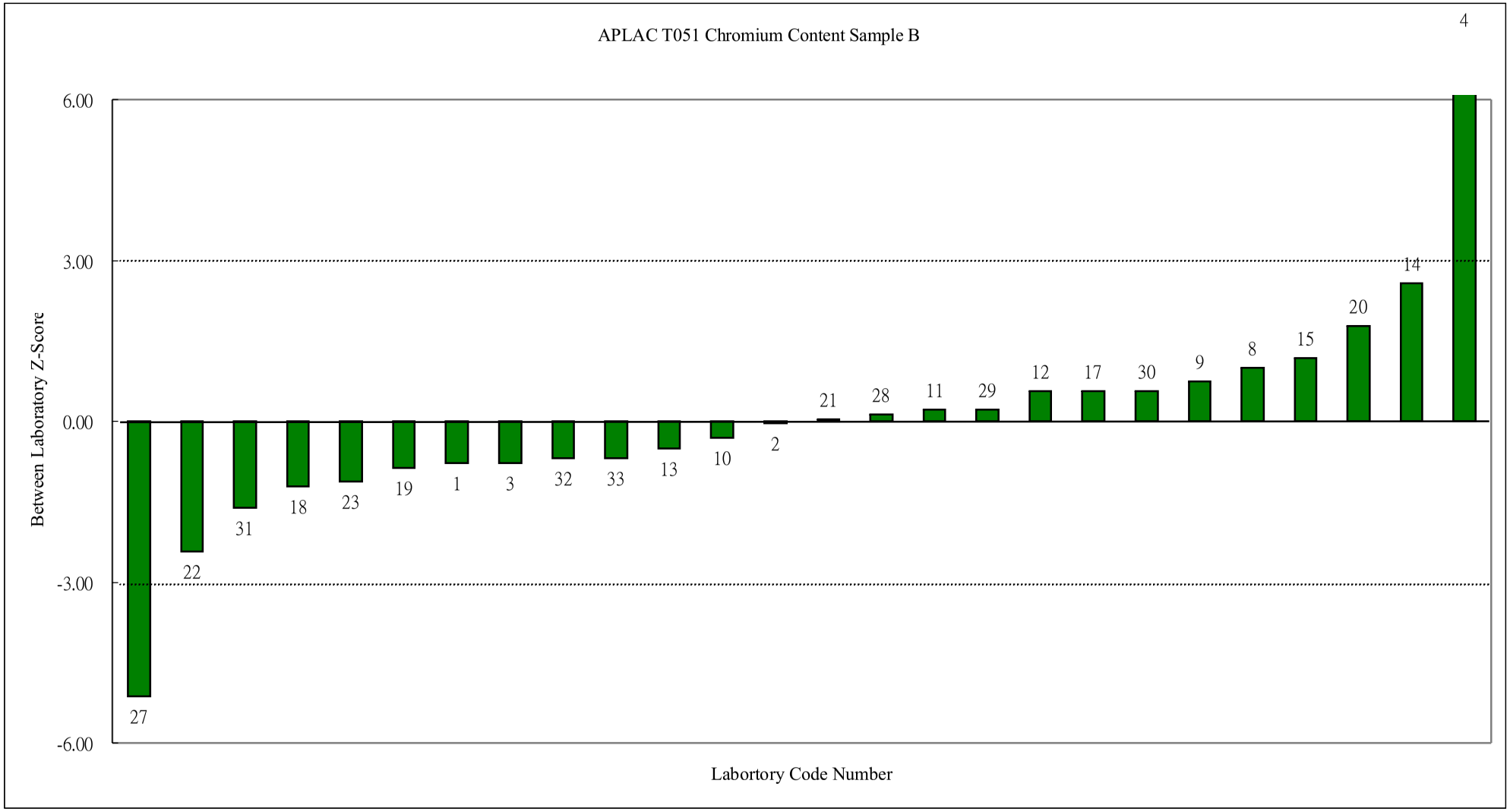
Lab Code	Result (ppm)	Z-Score	Expanded Uncertainty
1	15.5	-0.78	±0.2888
2	17.2	-0.04	0.036
3	15.5	-0.78	N/A
4	118.5	44.04※	±2.2
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	ND<2	N/A	N/A
8	19.6	1.00	N/A
9	19	0.74	±0.4
10	16.6	-0.30	±0.7
11	17.8	0.22	4.1
12	18.6	0.57	N/A
13	16.14	-0.50	1.90
14	23.2	2.57	0.0406
15	20	1.17	0.8
16	N/A	N/A	N/A
17	18.6	0.57	2.8
18	14.5	-1.22	30 (±U v%)
19	15.3	-0.87	25%
20	21.4	1.78	±29.8
21	17.4	0.04	3.2%
22	11.7	-2.44	2.1
23	14.7	-1.13	1.0
24	N/A	N/A	N/A
25	ND	N/A	N/A
26	<8	N/A	N/A
27	5.5	-5.13※	0.9
28	17.6	0.13	17%
29	17.8	0.22	±10%
30	18.6	0.57	16.8%
31	13.6	-1.61	3.2
32	15.7	-0.70	2.7
33	15.7	-0.70	1.57

No. of result	26
Median	17.3
Normalized IQR	2.30
Robust CV (%)	13.3
Maximum	118.5
Minimum	5.5
Range	113.0

NOTE:

1. ※ Denotes an outlier (i.e. | Z-Score| ≧ 3)
2. ND denotes not detect

A6.2


APLAC T051 Chromium Content Sample B
6.00
3.00
0.00
-3.00
-6.00
Between Laboratory Z-Score
Labortory Code Number
27
22
31
18
23
19
1
3
32
33
13
10
2
21
28
11
29
12
17
30
9
8
15
20
14
4

A7.1 Hg (A)

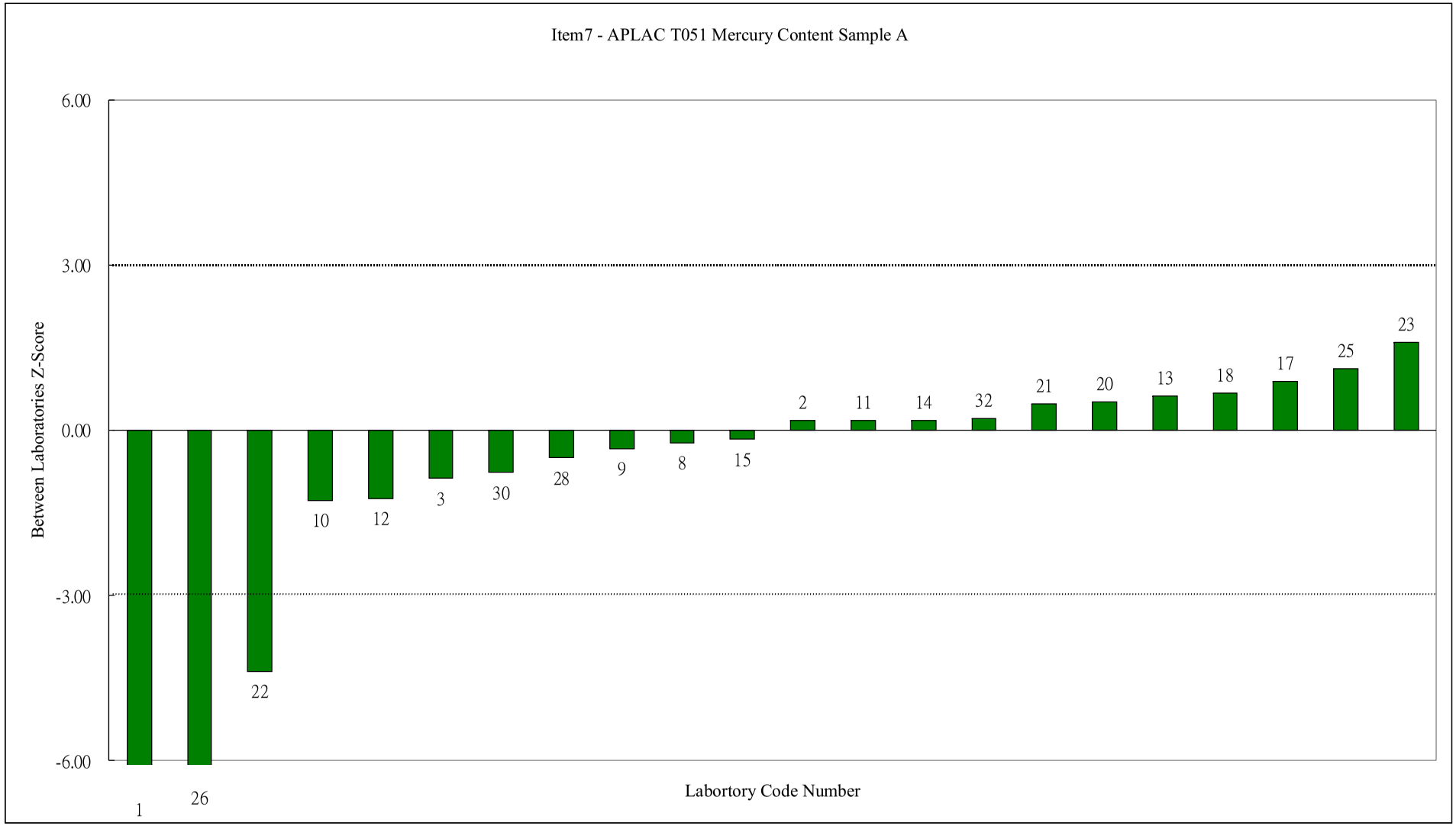
Lab Code	Result (ppm)	Z-Score	Expanded Uncertainty
1	0.5	-8.13※	±0.0586
2	25.1	0.17	0.048
3	22	-0.88	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	ND<10	N/A	N/A
8	23.9	-0.24	N/A
9	23.6	-0.34	±0.5
10	20.8	-1.28	±0.3
11	25.1	0.17	2.0
12	20.9	-1.25	N/A
13	26.4	0.61	2.04
14	25.1	0.17	0.0413
15	24.1	-0.17	5.0
16	N/A	N/A	N/A
17	27.2	0.88	2.6
18	26.6	0.67	30 (±U v%)
19	N/A	N/A	N/A
20	26.1	0.51	±9.0
21	26	0.47	1.8%
22	11.6	-4.38※	1.9
23	29.3	1.59	1.6
24	N/A	N/A	N/A
25	27.9	1.11	N/A
26	5.9	-6.31※	±1.3
27	N/A	N/A	N/A
28	23.1	-0.51	17%
29	<5	N/A	±10%
30	22.3	-0.78	13.6%
31	N/A	N/A	N/A
32	25.2	0.20	2.2
33	N/A	N/A	N/A

No. of result	22
Median	24.6
Normalized IQR	2.97
Robust CV (%)	12.1
Maximum	29.3
Minimum	0.5
Range	28.8

NOTE:

1. ※ Denotes an outlier (i.e. | Z-Score| ≧ 3)
2. ND denotes not detect

A7.2


Item7 - APLAC T051 Mercury Content Sample A
6.00
3.00
0.00
-3.00
-6.00
Between Laboratories Z-Score
1
26
22
10
12
3
30
28
9
8
15
2
11
14
32
21
20
13
18
17
25
23
Labortory Code Number

A8.1 Hg (B)

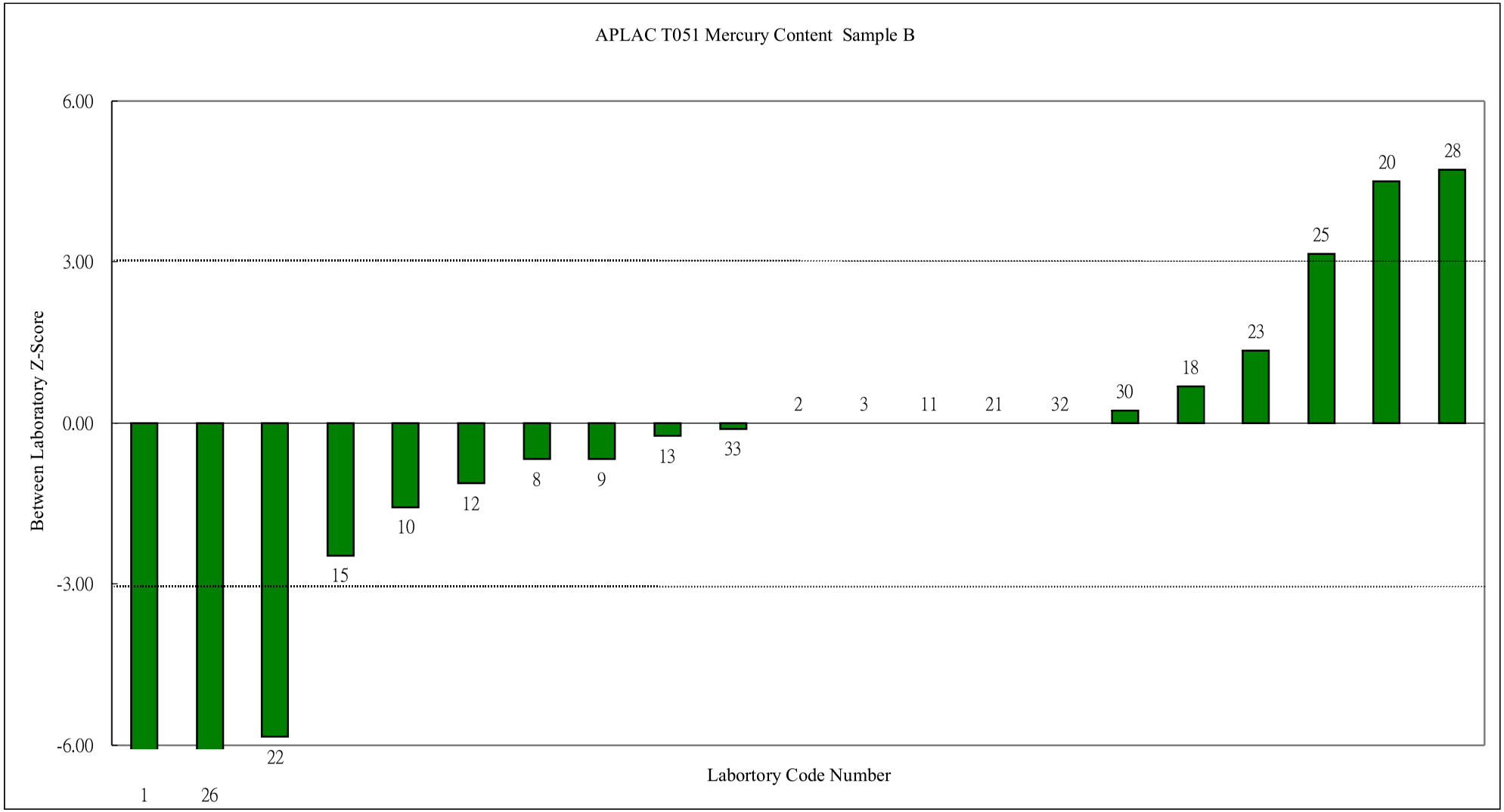
Lab Code	Result (ppm)	Z-Score	Expanded Uncertainty
1	0.3	-9.22※	±0.0270
2	4.4	0.00	0.048
3	4.4	0.00	N/A
4	N/A	N/A	N/A
5	N/A	N/A	N/A
6	N/A	N/A	N/A
7	ND<10	N/A	N/A
8	4.1	-0.67	N/A
9	4.1	-0.67	±0.5
10	3.7	-1.57	±1.0
11	4.4	0.00	2.0
12	3.9	-1.12	N/A
13	4.29	-0.25	2.04
14	ND	N/A	0.0413
15	3.3	-2.47	3.0
16	N/A	N/A	N/A
17	<10	N/A	1.1
18	4.7	0.67	30 (±U v%)
19	N/A	N/A	N/A
20	6.4	4.50※	±7.0
21	4.4	0.00	4.3%
22	1.8	-5.85※	0.5
23	5	1.35	0.3
24	N/A	N/A	N/A
25	5.8	3.15※	N/A
26	1.3	-6.97※	±0.4
27	N/A	N/A	N/A
28	6.5	4.72※	17%
29	<5	N/A	±10%
30	4.5	0.22	13.6%
31	N/A	N/A	N/A
32	4.4	0.00	0.3
33	4.35	-0.11	0.02

No. of result	21
Median	4.4
Normalized IQR	0.45
Robust CV (%)	10.1
Maximum	6.5
Minimum	0.3
Range	6.2

NOTE:

1. ※ Denotes an outlier (i.e. | Z-Score| ≧ 3)
2. ND denotes not detect

A8.2


APLAC T051 Mercury Content Sample B
6.00
3.00
0.00
-3.00
-6.00
Between Laboratory Z-Score
1
26
22
15
10
12
8
9
13
33
2
3
11
21
32
30
18
23
25
20
28
Labortory Code Number

A9.1

Test Methods

Lead Content

Lab Code	Method	Sample Preparation Method
1	EPA 6010B/ICP	Acid digestion
2	IEC62321 Ed.1	Microwave digestion
3	ICP-AES	Microwave digestion
4	ISO/IEC 62321 Ed1	Dry ashing and then acid treatment
5	N/A	N/A
6	Voltametry	Dry ashing
7	EPA 3051	Microwave digestion
8	AC/ROHS/0007 ver.1/05	Dry ashing/acid
9	EPA3052	Microwave
10	IEC62321 Ed.1, section11.4.2.3	1.microwave 2.ICP-OES
11	ICP-OES	Microwave digestion
12	EPA 6010B	EPA 3051A
13	IEC 62321	Microwave digestion
14	In-House method (RDMT-1602)	Microwave digestion
15	IEC 62321	Microwave
16	N/A	N/A
17	ICP	Acid microwave digestion
18	AAS (TNV 757467)	3ml H_2O_2+2ml HNO_3+2ml HCl
19	AAS flame1	Microwave digestion with HNO_3
20	X-ray EDS (energetic disperse spectrometry)	no
21	ICP-MS	Microwave digestion
22	ICP-AAS	Acid digestion
23	INAA	N/A
24	ICP-OES with ultrasonic nebulizer	Digestion acid with Pressure digestion system
25	IEC 62321, ED1	Dry ashing/acid
26	IEC 62321, ED1 AA FURNACE	Acid digestion
27	pr IEC 62321	Dry ashing
28	IEC 62321 Ed1/ CDV	Microwave
29	ICP and AAS	Acid digestion
30	ICP	Microwave digestion
31	AAS	Dry ashing/acid
32	AAS-ETA	Microwave digestion
33	IEC 62321 /1CD	Dry ashing/acid

A9.2

Test Methods

Cadmium Content

Lab Code	Method	Sample Preparation Method
1	EPA 6010B/ICP	Acid digestion
2	IEC62321 Ed.1	Microwave digestion
3	ICP-AES	Microwave digestion
4	ISO/IEC 62321 Ed1	Dry ashing and then acid treatment
5	N/A	N/A
6	Voltametry	Dry ashing
7	EPA 3051	Microwave digestion
8	AC/ROHS/0007 ver.1/05	Microwave digestion
9	EPA3052	Microwave
10	IEC62321 Ed.1, section11.4.2.3	1.microwave 2.ICP-OES
11	ICP-OES	Microwave digestion
12	EPA 6010B	EPA 3051A
13	IEC 62321	Microwave digestion
14	In-House method (RDMT-1602)	Microwave digestion
15	IEC 62321	Microwave
16	N/A	N/A
17	ICP	Acid microwave digestion
18	AAS-CSN EN ISO 5961(75 7418)	4ml $IHNO_3$+2ml HBF_4
19	AAS flame2)	Microwave digestion with HNO_3
20	X-ray EDS (energetic disperse spectrometry)	no
21	ICP-MS	Microwave digestion
22	ICP-AAS	Acid digestion
23	INAA	no
24	ICP-OES with ultrasonic nebulizer	Digestion acid with Pressure digestion system
25	IEC 62321, ED1	Dry ashing/acid
26	IEC 62321, ED1 AA FURNACE	Acid digestion modified: only HNO3
27	pr IEC 62321	Dry ashing
28	IEC 62321 Ed1/ CDV	Microwave
29	ICP and AAS	Acid digestion
30	ICP	Microwave digestion
31	AAS	Acid
32	AAS-ETA	Microwave digestion
33	IEC 62321 /1CD	Dry ashing/acid

A9.3

Test Methods

Chromium Content

Lab Code	Method	Sample Preparation Method
1	EPA 6010B/ICP	Acid digestion
2	IEC62321 Ed.1	Microwave digestion
3	ICP-AES	Microwave digestion
4	ISO/IEC 62321 Ed1	Dry ashing and then acid treatment
5	N/A	N/A
6	N/A	N/A
7	EPA 3060A	Microwave digestion
8	ICP-AES	Microwave digestion
9	EPA3052	Microwave
10	IEC 62321 Ed.1, section11.4.2.3	1.microwave 2.ICP-OES
11	ICP-OES	Microwave digestion
12	EPA 6010B	EPA 3051A
13	IEC 62321	Microwave digestion
14	In-House method (RDMT-1602)	Microwave digestion
15	IEC 62321	Microwave
16	N/A	N/A
17	ICP	Acid microwave digestion
18	AAS-CSN EN ISO 15586(75 7381)	4ml IHNO_3+2ml HBF_4
19	AAS-ETA	Microwave digestion with HNO_3
20	X-ray EDS (energetic disperse spectrometry)	no
21	ICP-MS	Microwave digestion
22	ICP-AAS	Acid digestion
23	INAA	no
24	N/A	N/A
25	IEC 62321, ED1	Alkaline Extraction
26	IEC 62321, ED1 VIS-540nm	Alkaline digestion
27	AAS	Dry ashing
28	IEC 62321 Ed1/ CDV	Microwave
29	ICP and AAS	Acid digestion
30	ICP	Microwave digestion
31	AAS	Acid
32	AAS-ETA	Microwave digestion
33	IEC 62321 /1CD	Dry ashing/acid

A9.4

Test Methods

Mercury Content

Lab Code	Method	Sample Preparation Method
1	EPA 6010B/ICP	Acid digestion
2	IEC62321 Ed.1	Microwave digestion
3	ICP-AES	Microwave digestion
4	N/A	N/A
5	N/A	N/A
6	N/A	N/A
7	EPA 3051	Microwave digestion
8	AC/ROHS/0007 ver.1/05	Microwave digestion
9	EPA3052	Microwave
10	IEC 62321 Ed.1, section10.4.3	1.microwave 2.ICP-OES
11	ICP-OES	Microwave digestion
12	EPA 6010B	EPA 3051A
13	IEC 62321	Microwave digestion
14	In-House method (RDMT-1602)	Microwave digestion
15	IEC 62321	Microwave
16	N/A	N/A
17	ICP	Acid microwave digestion
18	AMA 254 TNV 75 7440	N/A
19	N/A	N/A
20	X-ray EDS (energetic disperse spectrometry)	no
21	ICP-MS	Microwave digestion
22	ICP-AAS	Acid digestion
23	INAA	no
24	N/A	N/A
25	IEC 62321, ED1	Wet digestion
26	IEC 62321, ED1 CV-AAS	Acid digestion
27	N/A	N/A
28	IEC 62321 Ed1/ CDV	Microwave
29	ICP and AAS	Acid digestion
30	AAS	Microwave digestion
31	N/A	N/A
32	AAS-AMA254	Microwave digestion
33	IEC 62321 /1CD	Direct measurement

APPENDIX B

Preparation of the Samples

and

Homogeneity Testing

B1

PREPARATION OF THE SAMPLES

This sample was produced based on the certification of mass fraction of As, Br, Cd, Cl, Cr, Hg, Pb and S in two polyethylene CRMs (BCR-680 and BCR-681)

1. Matrix: PE powder
2. Process: Add the heavy metal drug with target concentration and the stabiliser to the base powder. After the melting extruder being cleaned and stable, add the desired mix powder to the material hopper for mix. Wait for 5 minutes after the colour of the mixture stabilises. Then, fetch the plastic sample and conduct tests.
3. Pattern of sample: plastic grains

HOMOGENEITY TESTING

Homogeneity tests had been made by PIDC before the samples were distributed to participants. Randomly select 15 samples out of 60 to determine the homogeneity of the samples in accordance with ASTM E826-85 (Re-approved 1996) *Standard practice for testing homogeneity of materials for development of reference materials*. A measurement was made on each of the 15 samples. The measurements were repeated four times. ASTM E826-85 methods were used to analyze the data.

CACULATIONS TO DETERMINE HOMOGENEITY

1. Compute T, B, t', and G, where: T = sum of each column; B = sum of each row; t' = mean of each column; and G = sum of $B_1 \ldots B_b$; b = number of replicate measurements (that is, runs); and t = number of specimens.
2. Choose a significance level (α) for the test.

   A 95% significance level is recommended for this procedure.

3. Obtain the q value that corresponds to t and v, where: v = the number of degrees of freedom:

$$v = (b-1) \times (t-1)$$

4. Compute S_t = sum of squares due to specimens:

$$S_t = [(T_1^2 + T_2^2 + T_3^2 + \cdots + T_t^2)/b] - (G^2/tb)$$

5. Compute S_b = sum of squares due to runs:

$$S_b = [(B_1^2 + B_2^2 + B_3^2 + \cdots + B_b^2)/t] - (G^2/tb)$$

6. Compute $\overline{S}$ = sum of the squares of all measurements in the table and subtractG^2/tb:

$$\overline{S} = (\sum_{i=1}^{t})(\sum_{j=1}^{b})Y_{ij}^2 - (G^2 / tb)$$

7. Compute:

$$s = \sqrt{(\overline{S} - S_b - S_t)/(b-1)(t-1)}$$

8. Compute:

$$w = qs/\sqrt{b}$$ [1]

9. If the absolute difference r_{ij} between any two mean values (that is, $t'1 ... t'n$) exceeds w, then there is strong evidence, at the 95% confidence level, that the specimens are not homogeneous. If the absolute difference r_{ij} between any two mean values does not exceed w, then the specimens shall be considered homogeneous. In this program, R means maximum of r_{ij}.

$$R = \underset{i,j}{Max}(r_{ij}), \quad \text{where } r_{ij} = \left|t_i' - t_j'\right| \ , \ i = 1,...,t \ , \ j = 1,...,b$$

or

$$R = \underset{i=1,...,t}{Max}(t_i') - \underset{i=1,...,t}{Min}(t_i')$$

[1] The q value is referred to Natrella, M.G., Experimental Statistics, NBS Handbook 91, U.S. Government Printing Office, Washington, DC, 1966.

B3

The Results of Homogeneity Sample A (RoHS)

b= 4 t= 15 mu= 42 q= 5.099

Pb	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	B(SUM)	B(SUM)^2
1	153.3	145.1	154.5	151.4	146.9	146.1	152.0	149.0	150.7	148.5	143.2	143.4	148.4	145.2	142.5	2220.2	4929288
2	154.7	149.3	147.3	143.9	150.5	147.9	149.3	152.5	150.5	149.3	150.8	154.3	152.4	150.4	158.2	2261.3	5113477.7
3	150.5	153.7	153.1	152.0	150.0	149.0	147.0	148.8	146.7	149.4	145.2	148.9	149.0	148.1	156.4	2247.8	5052604.8
4	148.9	147.0	151.6	140.1	152.6	146.9	151.7	147.9	155.9	151.3	150.6	145.2	149.1	142.6	144.4	2225.8	4954185.6
T(SUM)	607.400	595.1	606.5	587.4	600	589.9	600	598.2	603.8	598.5	589.8	591.8	598.9	586.3	601.5	8955.1	80193816
AVG	151.85	148.775	151.625	146.85	150	147.475	150	149.55	150.95	149.625	147.45	147.95	149.725	146.575	150.375	149.25167	
T(SUM)^2	368934.76	354144.01	367842.25	345038.76	360000	347982.01	360000	357843.24	364574.44	358202.25	347864.04	350227.24	358681.21	343747.69	361802.25		
1	23500.89	21054.01	23870.25	22921.96	21579.61	21345.21	23104	22201	22710.49	22052.25	20506.24	20563.56	22022.56	21083.04	20306.25		
2	23932.09	22290.49	21697.29	20707.21	22650.25	21874.41	22290.49	23256.25	22650.25	22290.49	22740.64	23808.49	23225.76	22620.16	25027.24		
3	22650.25	23623.69	23439.61	23104	22500	22201	21609	22141.44	21520.89	22320.36	21083.04	22171.21	22201	21933.61	24460.96		
4	22171.21	21609	22982.56	19628.01	23286.76	21579.61	23012.89	21874.41	24304.81	22891.69	22680.36	21083.04	22230.81	20334.76	20851.36		

St= 157.43733 Sb= 73.4805 S(mean)= 800.56983 S= 3.6828172 W= 9.3893426 MAX= 151.85 MIN= 146.575 R= 5.275 RSD= 2.4675217

Cd	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	B(SUM)	B(SUM)^2
1	131.3	129.0	136.3	139.6	133.2	130.7	138.3	138.9	136.0	136.9	129.5	137.4	132.3	135.3	139.8	2024.5	4098600.3
2	136.4	136.4	132.9	137.9	130.7	134.7	132.6	139.6	131.2	134.3	133.5	137.1	129.8	139.1	130.4	2016.6	4066675.6
3	141.1	133.6	144.1	144.5	141.9	127.4	141.1	135.6	144.2	126.7	136.3	137.2	136.8	134.6	132.6	2057.7	4234129.3
4	133.7	131.7	132.6	125.6	145.3	139.8	144.2	142.1	138.5	136.3	142.9	143.9	136.9	134.5	147.8	2075.8	4308945.6
T(SUM)	542.5	530.7	545.9	547.6	551.1	532.6	556.2	556.2	549.9	534.2	542.2	555.6	535.8	543.5	550.6	8174.6	66824085
AVG	135.625	132.675	136.475	136.9	137.775	133.15	139.05	139.05	137.475	133.55	135.55	138.9	133.95	135.875	137.65	136.24333	
T(SUM)^2	294306.25	281642.49	298006.81	299865.76	303711.21	283662.76	309358.44	309358.44	302390.01	285369.64	293980.84	308691.36	287081.64	295392.25	303160.36		
1	17239.69	16641	18577.69	19488.16	17742.24	17082.49	19126.89	19293.21	18496	18741.61	16770.25	18878.76	17503.29	18306.09	19544.04		
2	18604.96	18604.96	17662.41	19016.41	17082.49	18144.09	17582.76	19488.16	17213.44	18036.49	17822.25	18796.41	16848.04	19348.81	17004.16		
3	19909.21	17848.96	20764.81	20880.25	20135.61	16230.76	19909.21	18387.36	20793.64	16052.89	18577.69	18823.84	18714.24	18117.16	17582.76		
4	17875.69	17344.89	17582.76	15775.36	21112.09	19544.04	20793.64	20192.41	19182.25	18577.69	20420.41	20707.21	18741.61	18090.25	21844.84		

St= 259.81233 Sb= 155.29667 S(mean)= 1466.0273 S= 5.002186 W= 12.753073 MAX= 139.05 MIN= 132.675 R= 6.375 RSD= 3.6715088

R<W: Homogeneous

Remarks: 1. Sample styling: Particles

2. Test Instruments: XRF

3. Lab Condition: Temperature: 23±2℃, Relative Humidity: 50±5%

The Results of Homogeneity Sample A (RoHS)

b= 4 t= 15 mu= 42 q= 5.099

Hg	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	B(SUM)	B(SUM)^2
1	26.5	24.9	27.2	28.6	28.4	24.3	25.5	25.9	24.3	24.0	24.9	26.6	29.6	30.9	24.8	396.4	157132.96
2	30.0	28.3	29.1	25.5	22.1	27.0	28.7	27.6	26.2	21.6	28.3	21.1	26.8	27.7	27.2	397.2	157767.84
3	23.2	24.0	23.5	27.1	22.5	24.9	22.3	28.7	27.5	23.7	22.4	22.6	26.8	23.2	22.5	364.9	133152.01
4	28.5	22.3	23.4	30.7	21.9	30.7	26.6	22.8	24.8	26.0	30.7	21.7	26.7	28.7	26.0	391.5	153272.25
T(SUM)	108.2	99.5	103.2	111.9	94.9	106.9	103.1	105	102.8	95.3	106.3	92	109.9	110.5	100.5	1550	2402500
AVG	27.05	24.875	25.8	27.975	23.725	26.725	25.775	26.25	25.7	23.825	26.575	23	27.475	27.625	25.125	25.833333	
T(SUM)^2	11707.24	9900.25	10650.24	12521.61	9006.01	11427.61	10629.61	11025	10567.84	9082.09	11299.69	8464	12078.01	12210.25	10100.25		
1	702.25	620.01	739.84	817.96	806.56	590.49	650.25	670.81	590.49	576	620.01	707.56	876.16	954.81	615.04		
2	900	800.89	846.81	650.25	488.41	729	823.69	761.76	686.44	466.56	800.89	445.21	718.24	767.29	739.84		
3	538.24	576	552.25	734.41	506.25	620.01	497.29	823.69	756.25	561.69	501.76	510.76	718.24	538.24	506.25		
4	812.25	497.29	547.56	942.49	479.61	942.49	707.56	519.84	615.04	676	942.49	470.89	712.89	823.69	676		

St= 125.75833 Sb= 46.670667 S(mean)= 429.27333 S= 2.4729216 W= 6.3047136 MAX= 27.975 MIN= 23 R= 4.975 RSD= 9.5725998

Cr	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	B(SUM)	B(SUM)^2
1	139.3	111.2	116.8	112.0	170.1	180.0	111.4	146.0	131.2	187.3	150.9	136.6	140.5	123.9	127.3	2084.5	4345140.3
2	135.3	119.6	149.0	120.7	122.2	133.3	111.6	134.1	148.7	133.1	131.9	111.2	171.1	192.2	111.2	2025.2	4101435
3	114.9	111.2	158.2	111.2	133.1	120.9	156.5	134.3	141.8	134.5	136.1	111.2	111.2	115.4	123.4	1913.9	3663013.2
4	166.9	113.9	126.5	116.7	111.2	111.2	125.6	133.7	150.5	141.5	140.1	111.2	111.2	122.3	121.6	1904.1	3625596.8
T(SUM)	556.4	455.9	550.5	460.6	536.6	545.4	505.1	548.1	572.2	596.4	559	470.2	534	553.8	483.5	7927.7	62848427
AVG	139.1	113.975	137.625	115.15	134.15	136.35	126.275	137.025	143.05	149.1	139.75	117.55	133.5	138.45	120.875	132.12833	
T(SUM)^2	309580.96	207844.81	303050.25	212152.36	287939.56	297461.16	255126.01	300413.61	327412.84	355692.96	312481	221088.04	285156	306694.44	233772.25		
1	19404.49	12365.44	13642.24	12544	28934.01	32400	12409.96	21316	17213.44	35081.29	22770.81	18659.56	19740.25	15351.21	16205.29		
2	18306.09	14304.16	22201	14568.49	14932.84	17768.89	12454.56	17982.81	22111.69	17715.61	17397.61	12365.44	29275.21	36940.84	12365.44		
3	13202.01	12365.44	25027.24	12365.44	17715.61	14616.81	24492.25	18036.49	20107.24	18090.25	18523.21	12365.44	12365.44	13317.16	15227.56		
4	27855.61	12973.21	16002.25	13618.89	12365.44	12365.44	15775.36	17875.69	22650.25	20022.25	19628.01	12365.44	12365.44	14957.29	14786.56		

St= 6492.7743 Sb= 1538.5658 S(mean)= 24679.602 S= 19.909475 W= 50.759206 MAX= 149.1 MIN= 113.975 R= 35.125 RSD= 15.068286

R<W: Homogeneous

Remarks: 1. Sample styling: Particles
2. Test Instruments: XRF
3. Lab Condition: Temperature: 23±2℃, Relative Humidity: 50±5%

B5

The Results of Homogeneity Sample B (RoHS)

b= 4 t= 15 mu= 42 q= 5.099

Pb	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	B(SUM)	B(SUM)^2
1	21.5	17.6	16.8	17.4	19.2	15.3	18.3	20.7	15.4	18.3	20.8	14.2	15.8	18.3	14.9	264.5	69960.25
2	19.1	17.6	15.4	19.7	20.5	21.7	21.1	17.8	22.4	17.1	15.2	19.4	17.1	16.5	17.4	278	77284
3	18.6	18.5	22.1	18.3	18.1	19.9	18.8	17.8	18.9	18.6	22.6	19.3	18.0	18.1	20.3	287.9	82886.41
4	18.6	18.9	19.5	16.5	17.3	21.5	22.8	18.4	18.2	19.4	20.8	21.4	19.8	19.2	20.6	292.9	85790.41
T(SUM)	77.8	72.6	73.8	71.9	75.1	78.4	81	74.7	74.9	73.4	79.4	74.3	70.7	72.1	73.2	1123.3	1261802.9
AVG	19.45	18.15	18.45	17.975	18.775	19.6	20.25	18.675	18.725	18.35	19.85	18.575	17.675	18.025	18.3	18.721667	
T(SUM)^2	6052.84	5270.76	5446.44	5169.61	5640.01	6146.56	6561	5580.09	5610.01	5387.56	6304.36	5520.49	4998.49	5198.41	5358.24		
1	462.25	309.76	282.24	302.76	368.64	234.09	334.89	428.49	237.16	334.89	432.64	201.64	249.64	334.89	222.01		
2	364.81	309.76	237.16	388.09	420.25	470.89	445.21	316.84	501.76	292.41	231.04	376.36	292.41	272.25	302.76		
3	345.96	342.25	488.41	334.89	327.61	396.01	353.44	316.84	357.21	345.96	510.76	372.49	324	327.61	412.09		
4	345.96	357.21	380.25	272.25	299.29	462.25	519.84	338.56	331.24	376.36	432.64	457.96	392.04	368.64	424.36		

St= 31.2 Sb= 31.4 S(mean)= 242.3 S= 2.1 W= 5.3 MAX= 20.3 MIN= 17.7 R= 2.6 RSD= 11.1

Cd	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	B(SUM)	B(SUM)^2
1	19.5	16.7	19.3	17.8	21.0	22.1	19.4	22.4	17.6	19.5	21.1	23.5	20.8	18.7	19.8	299.2	89520.64
2	17.9	24.3	19.4	20.3	22.5	18.5	20.7	18.0	24.1	21.8	21.6	16.9	17.0	18.1	16.8	297.9	88744.41
3	19.5	18.2	21.0	16.0	24.8	15.9	16.1	21.5	24.5	20.1	16.9	18.4	15.6	16.4	25.2	290.1	84158.01
4	20.1	19.9	24.4	24.3	19.9	16.8	22.7	23.3	18.7	22.3	18.5	25.4	21.8	21.9	20.9	320.9	102976.81
T(SUM)	77	79.1	84.1	78.4	88.2	73.3	78.9	85.2	84.9	83.7	78.1	84.2	75.2	75.1	82.7	1208.1	1459505.6
AVG	19.25	19.775	21.025	19.6	22.05	18.325	19.725	21.3	21.225	20.925	19.525	21.05	18.8	18.775	20.675	20.135	
T(SUM)^2	5929	6256.81	7072.81	6146.56	7779.24	5372.89	6225.21	7259.04	7208.01	7005.69	6099.61	7089.64	5655.04	5640.01	6839.29		
1	380.25	278.89	372.49	316.84	441	488.41	376.36	501.76	309.76	380.25	445.21	552.25	432.64	349.69	392.04		
2	320.41	590.49	376.36	412.09	506.25	342.25	428.49	324	580.81	475.24	466.56	285.61	289	327.61	282.24		
3	380.25	331.240	441	256	615.04	252.81	259.21	462.25	600.25	404.01	285.61	338.56	243.36	268.96	635.04		
4	404.01	396.01	595.36	590.49	396.01	282.24	515.29	542.89	349.69	497.29	342.25	645.16	475.24	479.61	436.81		

St= 69.619 Sb= 34.897833 S(mean)= 422.0965 S= 2.7498037 W= 7.0106246 MAX= 22.05 MIN= 18.325 R= 3.725 RSD= 13.656835

R<W: Homogeneous

Remarks: 1. Sample styling: Particles

2. Test Instruments: XRF

3. Lab Condition: Temperature: 23±2℃, Relative Humidity: 50±5%

B6

The Results of Homogeneity Sample B (RoHS)

b= 4 t= 15 mu= 42 q= 5.099

Hg	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	B(SUM)	B(SUM)^2
1	5.0	9.4	8.4	6.2	8.8	3.7	5.9	3.0	7.3	6.7	5.2	7.5	5.9	4.7	4.5	92.2	8500.84
2	5.8	8.8	5.8	8.2	6.7	3.6	4.7	3.9	5.7	3.4	8.6	4.7	4.0	6.3	9.5	89.7	8046.09
3	4.0	6.8	4.3	3.6	4.9	8.1	5.8	3.3	8.5	3.3	7.2	3.6	8.6	3.8	4.4	80.2	6432.04
4	9.6	4.7	4.7	7.3	6.5	3.7	9.6	7.2	3.9	6.4	6.6	4.2	8.1	3.9	9.5	95.9	9196.81
T(SUM)	24.4	29.7	23.2	25.3	26.9	19.1	26	17.4	25.4	19.8	27.6	20	26.6	18.7	27.9	358	128164
AVG	6.1	7.425	5.8	6.325	6.725	4.775	6.5	4.35	6.35	4.95	6.9	5	6.65	4.675	6.975	5.9666667	
T(SUM)^2	595.36	882.09	538.24	640.09	723.61	364.81	676	302.76	645.16	392.04	761.76	400	707.56	349.69	778.41		
1	25	88.36	70.56	38.44	77.44	13.69	34.81	9	53.29	44.89	27.04	56.25	34.81	22.09	20.25		
2	33.64	77.44	33.64	67.24	44.89	12.96	22.09	15.21	32.49	11.56	73.96	22.09	16	39.69	90.25		
3	16	46.24	18.49	12.96	24.01	65.61	33.64	10.89	72.25	10.89	51.84	12.96	73.96	14.44	19.36		
4	92.16	22.09	22.09	53.29	42.25	13.69	92.16	51.84	15.21	40.96	43.56	17.64	65.61	15.21	90.25		

St= 53.3 Sb= 9.0 S(mean)= 234.6 S= 2.0 W= 5.2 MAX= 7.4 MIN= 4.4 R= 3.1 RSD= 33.9

Cr	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	B(SUM)	B(SUM)^2
1	10.1	10.1	9.8	12.4	11.7	9.4	12.3	11.9	11.4	8.2	8.9	9.3	11.2	10.1	9.6	156.4	24460.96
2	9.7	8.8	12.2	8.8	11.5	10.8	12.1	10.1	9.7	8.7	12.5	9.9	9.4	12.5	11.6	158.3	25058.89
3	11.8	11.3	5.9	10.1	8.7	11.1	9.8	11.2	11.7	11.1	11.9	8.6	9.5	8.7	10.3	151.7	23012.89
4	9.1	10.1	8.4	9.6	12.4	12.2	11.3	8.0	9.3	10.5	9.1	9.9	9.9	11.8	11.7	153.3	23500.89
T(SUM)	40.7	40.3	36.3	40.9	44.3	43.5	45.5	41.2	42.1	38.5	42.4	37.7	40	43.1	43.2	619.7	384028.09
AVG	10.175	10.075	9.075	10.225	11.075	10.875	11.375	10.3	10.525	9.625	10.6	9.425	10	10.775	10.8	10.328333	
T(SUM)^2	1656.49	1624.09	1317.69	1672.81	1962.49	1892.25	2070.25	1697.44	1772.41	1482.25	1797.76	1421.29	1600	1857.61	1866.24		
1	102.01	102.01	96.04	153.76	136.89	88.36	151.29	141.61	129.96	67.24	79.21	86.49	125.44	102.01	92.16		
2	94.09	77.44	148.84	77.44	132.25	116.64	146.41	102.01	94.09	75.69	156.25	98.01	88.36	156.25	134.56		
3	139.24	127.69	34.81	102.01	75.69	123.21	96.04	125.44	136.89	123.21	141.61	73.96	90.25	75.69	106.09		
4	82.81	102.01	70.56	92.16	153.76	148.84	127.69	64	86.49	110.25	82.81	98.01	98.01	139.24	136.89		

St= 22.299333 Sb= 1.7738333 S(mean)= 117.70183 S= 1.4930686 W= 3.8065785 MAX= 11.375 MIN= 9.075 R= 2.3 RSD= 14.456046

R<W: Homogeneous

Remarks: 1. Sample styling: Particles

2. Test Instruments: XRF

3. Lab Condition: Temperature: 23±2℃, Relative Humidity: 50±5%

The results of homogeneity testing indicated the samples suitable for use in this proficiency testing program. Thus any outlier results subsequently identified in this program will not be attributable to samples variability.

APPENDIX C

Instructions to Participants

Results Sheet

Measurement Uncertainty

C1

Asia Pacific Laboratory Accreditation Cooperation

APLAC T051 Heavy Metal Elements in Plastic Proficiency Testing Program

INSTRUCTIONS TO PARTICIPATING LABORATORIES

To ensure that results from this program can be analysed properly, participants are asked to adhere carefully to the following instructions.

1. SAMPLE

Each participating laboratory is given two sealed amber bottle containing about 7 g plastic particle sample with labelled APLAC T051 A#, APLAC T051 B#.

Upon receipt, unpack the sample and inspect them for any damage. If any, please contact your accreditation body.

2. TESTS TO BE PERFORMED

Each participating laboratory is required to determine the level (ppm or mg/kg) of Lead Content, Cadmium Content, Chromium Content and Mercury Content in sample A and sample B.
Storage Detail: Avoid direct exposure to sunlight.
Testing Period: Testing should be finished **within two week** after receiving the samples.
Method used: Participating laboratories are recommended to use IEC 62321, Ed.1: Procedures for the Determination of Regulated Substances in Electrotechnical Products or method for which accreditation is held and record the method used on the result sheet.

3. DOCUMENTS TO BE SUBMITTED

Within **two week** of the completion of the tests, participating laboratories are required to send result sheet to their accreditation body:

No other documents are required. Laboratories should make a copy of all documents and worksheets for their own records and keep these on file for an adequate period of time.

A final report will be issued at the end of the program with each laboratory only identified by a **confidential** code number.

4. CONFIDENTIALITY

For this program your laboratory will be assigned a code number after TAF received result sheet. All reference to your laboratory in reports associated with this program will be with this code number, thus ensuring confidentiality of results.

5. GENERAL INFORMATION

For general queries, please contact your accreditation body.

Additional information may be obtained from:

Ms. Jean Yang
Taiwan Accreditation Foundation
TEL： +886-3-5714848 ext 224
FAX：+886-3-5726308
E-mail: jeanyang@taftw.org.tw

Mr. Hung-Chang Huang
Taiwan Accreditation Foundation
TEL： +886-3-5714848 ext 208
E-mail: hchuang@taftw.org.tw

C3

Asia Pacific Laboratory Accreditation Cooperation

APLAC T051 Heavy Metal Elements in Plastic Proficiency Testing Program
RESULTS SHEET

Please complete this results sheet and return it to your national accreditation body and TAF.

Lab Code:
(Assigned after test)

Accreditation body:

_____

Testing laboratory: _____

Date of test： _____

Summary of Test Results

Item(s)	Result		Uncertainty	Test Method	Sample Preparation Method (Dry ashing/acid or microwave digestion)
	Sample A No.:___.	Sample B No.:___.			
Lead Content					
Cadmium Content					
Chromium Content					
Mercury Content					
Remark(s): 1.Unit: ppm (mg/Kg) 2.Report all test to 1 decimal precision 3.Average Weight of Sample:					

Signature： _____ **Date：** _____

C4

Measurement Uncertainty Budget

Items	Factors of Uncertainty x_i	Type A / Type B	Probability Distribution	Standard Uncertainty $u(x_i)$	Coverage Fact or(F
Combined Standard Uncertainty $u_c(Y)$ **:**					
Expanded Uncertainty (95% confidence interval) $U_{exp}=\kappa=\kappa_c(y)$ **:**					

Note：Please participating laboratories provide test original data and e-mail or fax to TAF.

Signature： _____ **Date：** _____

C5

INSTRUCTIONS – MEASUREMENT UNCERTAINTY
Part (1) Background information & justification for this change

ISO/IEC 17025 requires that, except under specified conditions, the uncertainty of measurement associated with the results of tests and measurements must be estimated.

What is uncertainty of measurement?
Uncertainty of measurement is defined as a "parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measured" (the measured is the particular quantity subject to measurement).

The result of a test or measurement is our best estimate of the true value of the measured. The result is imperfect. The true value of the measured is contained within a range of values about the measurement result and the "uncertainty of measurement" is an estimate of the magnitude of that range expressed at a given level of confidence (confidence interval). Uncertainty of measurement is usually given as a 95 % confidence interval and would normally be expressed in the appropriate SI units (i.e. mm, °C, g/l, MPa etc).

For example, the result of a measurement might be 5.1 mg/l with an uncertainty of ±0.2 mg/l at a 95 % level of confidence. This means that there is an estimated 95 % probability that the true value is in the range 4.9 mg/l to 5.3 mg/l. The 95 % probability means that there is an estimated one in twenty chance that the true value is outside that range.

Uncertainty of measurement may also be expressed as a percentage where appropriate.

To assist laboratories to comply with the requirements of ISO/IEC 17025 for estimating uncertainty and to promote a uniform methodology in its estimation, information packages for APLAC PT testing program participants now include general guidance relating to estimating uncertainties for the specific testing involved, and final program reports will now include relevant worked examples. All program participants are required to report their estimates of uncertainty to their accreditation bodies along with their results unless the Technical Adviser to the program specifically waives any requirement to estimate uncertainties. The estimates of uncertainty provided by participants will be incorporated into the final program reports enabling direct comparison of uncertainty estimates across the program participants. The uncertainty estimates will not be used in the evaluation of the results on the primary samples.

C6

How is uncertainty of measurement to be estimated?

APLAC expects that program participants' uncertainties of measurement would be estimated in accordance with the requirements of the respective member accreditation bodies. There are different approaches and methodologies available. Worked examples provided in APLAC PT program reports will generally be based on ISO GUM but will recognise other methodologies in accordance with 5.4.6.3 NOTE 3 in ISO 17025.

Estimates of uncertainty of measurement provided by program participants are required to be given at the 95 % level of confidence.

ISO GUM methodology

An estimate of uncertainty of measurement would usually be based on the combination of a number of influencing parameters (components of uncertainty) such as errors in reference values, instrument errors, repeatability, thermal effects, weighing errors, in homogeneity etc. ISO GUM methodology requires that the influence of each component of uncertainty on the measurement result be quantified and expressed numerically as a standard deviation. These values are then combined according to the rules of the propagation of uncertainty to produce a combined standard deviation (combined standard uncertainty) and the combined standard uncertainty is multiplied by a coverage factor to produce an expanded uncertainty at the required level of confidence. Detailed descriptions and information on the implementation of this methodology have been published by ISO2, UKAS3 and Eurachem/CITAC4 and made available over the internet[7].

Uncertainty of measurement is best estimated within the individual laboratory environment. All factors which will have a significant influence on the test or measurement result must be included in the estimation process. There must be suitable programs utilising reference standards, instruments and materials to ensure on-going and adequate quality control and repeatability and reproducibility of methods and equipment over time. In many instances, it will be possible to utilise quality control data in assessing uncertainty components such as precision. Where these data are not available, it may be necessary to carry out precision studies or to rely on published information about the method or portions of it until the laboratory can obtain its own estimates.

APLAC is aware of the general need for better estimates of uncertainty, and estimates that are obtained under similar conditions in all laboratories. PT programs are useful mechanisms for spreading awareness of uncertainty of measurement and the effects of different ways of estimating it. We anticipate that the information made available through PT programs will help focus discussions on uncertainty of measurement.

APLAC Technical Committees will interpret the information and report on current practices. They will also make recommendations for improving the collection of uncertainty data, the estimation of uncertainties and incorporating data and information on uncertainty of measurement into PT program reports. Therefore we anticipate an evolution in the mechanisms for collecting and reporting uncertainty data and associated information over the next few years.

Participation in APLAC PT programs should assist laboratories to develop appropriate estimates of uncertainty, help to guide accreditation bodies to adopting common and consistent approaches leading to enhanced understanding and international comparability of measurements among the member nations.

APLAC will consider the use of estimates of uncertainty of measurement in the evaluation of its PT testing program results after it is satisfied that participating laboratories are estimating uncertainties of measurement in an appropriate and consistent manner.

Here are a few important terms:

Standard uncertainty (*u(xi)*) is an input component of uncertainty *xi* expressed as a standard deviation. It should be expressed in the units of the influencing parameter, but may be expressed as a percentage where convenient.

Type A evaluation estimates of standard uncertainty are evaluated by applying statistical techniques to a series of repeatability or curve fitting data. For example, a standard uncertainty estimated from the repeatability of measurements on replicate samples is a Type A evaluation.

Type B evaluation estimates of standard uncertainty are based on assumed probability distributions, experience, laboratory records, or other information. For example, a standard uncertainty estimated using data provided on a calibration certificate is a Type B evaluation.

Sensitivity coefficient (*ci*) is the mathematical relationship between an influencing parameter and its effect on the result of a measurement. In many instances it is unity. That is, there is a one to one relationship between the value of the influence and its effect on the measurement result. For example, when weighing a sample of material, any uncertainty due to errors in the balance reading will have a one to one effect on the measurement result. On the other hand, if we are considering the influence of temperature on the length of a metal bar then the sensitivity coefficient is equal to the coefficient of linear thermal expansion for the metal bar multiplied by the length of the bar. It is important to note that a sensitivity coefficient has units. It is also important to note that the calculation methodology used by Eurachem/CITAC4 incorporates sensitivity coefficients in a manner that does not require their specific evaluation.

Combined standard uncertainty (*uc(y)*) is the final estimate of uncertainty for the test or measurement result *y* expressed as a standard deviation. It is calculated by multiplying the standard uncertainty *u(xi)* for each input component *(xi)* with its respective sensitivity coefficient *ci* to produce *ciu(xi)* and then combining those values by taking the square root of the sum of their squares. Note that the products *ciu(xi)* must each be expressed in the same units as those required for expressing the combined estimate *uc(y)*.

Expanded uncertainty (*U*) is the final result of our estimate of uncertainty expressed as a confidence interval. It is calculated by multiplying the combined standard uncertainty by a coverage factor to produce the desired level of confidence (usually 95 %).

Coverage factor (*k*) is a multiplier used to expand the combined standard uncertainty *uc(y)* to an interval that is estimated to contain the true value of the measured at a given level of confidence ($U = k.uc(y)$). The coverage factor then represents the number of standard deviations in the expanded uncertainty and is determined according to the Student-t distribution. A coverage factor of 2 is commonly used to approximate the expanded uncertainty to the 95 % confidence level.

References:

1. ISO-IEC 17025:1999. General requirements for the competence of testing and calibration laboratories. ISO, Geneva (1999)
2. Guide to the Expression of Uncertainty in Measurement. ISO, Geneva (1993)
3. UKAS LAB 12: The Expression of Uncertainty in Testing. UKAS, London (2000)
4. Eurachem / CITAC Guide QUAM: 2000.P1. Quantifying Uncertainty in Analytical Measurement, 2nd Edition (2000)
5. ISO/DTS 21748:2002 Guide for the use of repeatability, reproducibility, and trueness estimates in measurement uncertainty estimation.
6. APLAC TC 005, Interpretation and Guidance on the Estimation of Uncertainty of Measurement in Testing
7. Websites: www.A2LA.org / (for A2LA policies, links to guidance documents, including the UKAS Guide, and the Eurachem/ CITAC Guide, at no cost) www.measurementuncertainty.org / (Eurachem / CITAC Guide) www.fasor.com /iso25/ (general information, links, and discussion of ISO-IEC 17025).

APPENDIX D

Statistical Procedures
Calculations and Formulae

STATISTICAL PROCEDURES

For each statistically analysed test, the following information is given:

(a) a table of results and calculated Z-Scores;

(b) a list of summary statistics; and

(c) ordered Z-Score charts;

Table of Results and Robust Z-Scores

These tables contain the results returned by each laboratory, and the Z-Scores calculated for each pair of results.

Please note that the results are presented in the table "as reported" by participants.

Outliers are identified in the table by a marker (※) next to the relevant Z-Scores.

Summary Statistics

The list of summary statistics appears at the bottom of the table of results and consists of:

1. The number of results for that test/sample (*No. of Results*);
2. The median of laboratory's results - i.e. the middle value (*Median*);
3. The normalised interquartile range of the results (*Normalised IQR*) – the interquartile range times 0.7413;
4. The robust coefficient of variation, expressed as a percentage (*Robust CV*) - i.e.

   $100 \times \textit{Normalised IQR} \div \textit{Median}$;
5. The minimum and maximum laboratory results; and
6. The range (*Maximum* - *Minimum*).

Ordered Z-Score Charts

On these charts each laboratory's Z-Score is shown, in order of magnitude, and is marked with its code number. From these each laboratory can readily compare its performance relative to the other laboratories.

These charts contain solid lines at +3 and –3, so the outliers are clearly identifiable as the laboratories whose "bar" extends beyond these "cut off" lines. The y-axis has been limited to range from –6 to +6, so in some cases very large or small (negative) Z-Scores appear as extending beyond the limit of the chart.

STATISTICAL CALCULATIONS AND FORMULAE

Robust Z-Scores are calculated by replacing the mean and standard deviation in the "classical" Z-Score by the median and normalised IQR, respectively - i.e. the Z-Score for a result is the result minus the median (all) divided by the normalised IQR.

$$\text{Z-Score} = \frac{\text{Result} - Median}{NormalizedIQR}$$

The median is the middle value of the group, i.e. half of the results are higher than it and half are lower. It is calculated from the sorted values (from lowest to highest). If *N* is an odd number, the median is the singular central value. If *N* is even, it is the average of the two central values.

The interquartile range (*IQR*) is the difference between the lower and upper quartiles. The lower quartile (*Q1*) is the value *below that* a quarter of the results laid. Similarly, the upper quartile (*Q3*) is the value *above* that a quarter of the results laid. The quartiles are calculated analogously to the median and $IQR=Q3-Q1$. The "Normalised IQR" equals $IQR \times 0.7413$. The factor 0.7413 comes from the standard normal distribution, which has a mean of zero and a standard deviation equal to one. The width of the interquartile range of such a distribution is 1.34898 and 1/1.34898 = 0.7413.

Multiplying the *IQR* by this factor makes it comparable to a standard deviation. The "Robust CV" is a coefficient of variation and is equal to the normalised IQR divided by the median, expressed as a percentage (i.e. multiplied by 100). The minimum, maximum and range are the lowest value, the highest value and the difference between them (respectively).