



APLAC

Asia Pacific Laboratory Accreditation Cooperation

APLAC T070 Determination of PBDE in Acrylonitrile butadiene styrene (ABS) Proficiency Testing Program

Final Report

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1. Introduction

This report summarizes the results of APLAC T070 **Determination of PBDE in Acrylonitrile butadiene styrene (ABS)** Proficiency Testing Program involving testing laboratories for the Asia Pacific Laboratory Accreditation Cooperation (APLAC).

2. Program Feature

- (a) APLAC, EA and ILAC members were invited to participate. There were in total 35 laboratory participants nominated by 12 accreditation bodies (ABs) in this program. All samples were sent to the ABs and transferred to the participating laboratories.

Table 1 Summary of Economies and Participants

Economy	No. of laboratories nominated	No. of laboratories returning result
Australia	1	0
China	4	4
Hong Kong	4	4
Japan	7	6
Korea	2	0
Malaysia	4	3
MEXICO	1	0
Singapore	3	3
Taiwan	4	4
Thailand	4	4
USA	1	1
Total	35	29

- (b) Participating laboratories were supplied two amber sample bottles. Each bottle contains about 7 gram of ABS pellet.
- (c) Each participating laboratory is required to analysis the concentration of **PBDE, BDE-196, BDE-197, BDE-203**.
- (d) 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 samples. (Appendix C)
- (e) 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 Appendices

APPENDIX A contains:

- (a) The results of samples reported by participants and the calculated Z-Score result for each test on each sample.
- (b) A table of robust statistics for each test, such as number of results, median, normalized 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 laboratory.

APPENDIX B contains the 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 formula.

4. Statistical design of the program

A split level statistical design was used in this program. The samples labeled Sample A, and Sample B (sample pair) was identical.

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

For each laboratory, the between-laboratories z-score was calculated for the sample pair. This was based on the (standardized) sum of the calculated mean of the duplicate or triplicate results reported for each sample. The within laboratory z-score was based on the (standardized) difference between the mean of the duplicate or triplicate results reported for each sample.

For each laboratory, a single robust Z-score was calculated for each returned result.

For each item, we can use the Z-score of each laboratory to understand the degree that the result of that laboratory deviates from the population. If $|Z - \text{Score}| \geq 3$ it represents that the testing result is an outlier and we will denote it by “✕”. For the testing result that $|Z - \text{Score}| \geq 3$ one should make further assessment.

5. Outlier Results

The outliers of feedback results are shown as below, the detail information please refers to the Summary of Results in Appendix A.

Table 2 List of Outliers

$ Z\text{-Score} \geq 3$	Lab Code	Proportion (%)
Sample A		
PBDE	23	3.4%
BDE-196	14	3.4%
BDE-197	14	3.4%
BDE-203	23,26,27	10.3%
Sample B		
PBDE	23	3.4%
BDE-196	14	3.4%
BDE-197	14	3.4%
BDE-203	23,26,27	10.3%

6. Technical Comments

In this brominated flame retardant proficiency test, there are 35 laboratory registered to participate. The participant laboratories and their nationality are listed in Table 1. After completing registration procedure, each participant has received two test samples. Three concentrations of octa-brominated biphenyl ether congeners (BDE-196, BDE-197 and BDE-203) in test samples are reported from participant laboratories. All test results summary of participant laboratories and their statistics are listed in Table 4. The histograms of Z-score of test results are listed in Appendix A.

When population is a normal distribution, the distribution ratio is 99.73% which absolute value of Z score is below 3. The Z score could be estimated the deviation degree between test results and the mean of the sample. The abnormality of inter-laboratory and within-laboratory could be evaluated by ZB and ZW. When ZB is above 3 or below -3, it shows that there is a systematic error existed in laboratory. When ZW is above 3 or below -3, it shows that there is a random error existed in laboratory. If the test result deviates from the mean of the sample in 1st and 3rd quadrant, there is a random error existed in laboratory. If the test result deviates from the mean of the sample in 2nd and 4th quadrant, there is a random error existed in laboratory.

Appendix A

Summary of Results

Information of Test

Table 3 Laboratories' Result for PBDE, BDE-196, BDE-197, BDE-203

Item 1	Sample A : PBDE					
Lab Code	Result (mg/kg)				Spike Recovery (%)	Expanded Uncertainty (mg/kg)
1	-	-	-	-	-	-
2	-	-	-	-	-	-
3	-	-	-	335.869	-	24.097
4	-	-	-	-	-	-
5	212	201	-	-	94.9	-
6	241	-	-	-	-	-
7	-	-	-	-	-	-
8	241.05	-	-	-	81.57	0.03
9	-	-	-	-	-	-
10	-	-	-	-	-	-
11	329	328	324	-	-	2.6
12	249	253	-	251	112 / 112	±58.0
13	67.0	68.8	70.7	68.9	93.8 / 96.9 / 97.1 / 95.9	-
14	322	296	-	-	87.2 / 81.2	19.9
15	270	270	-	-	83.5 / 87.4	50
16	380	382	-	381	-	76.2
17	331	270	282	294	-	64.7
18	339	363	-	351	-	17.0
19	-	-	-	-	-	-
20	-	-	-	-	-	-
21	-	-	-	-	-	-
22	-	-	-	-	-	-
23	642	-	-	-	88	-
24	167.166	-	-	-	118.4	±31
25	-	-	-	-	-	-
26	-	-	-	-	-	-
27	-	-	-	-	-	-
28	360	381	373	371	-	±64
29	238	239	256	244	90	±49
30	114.8	105.8	101.3	107.3	-	-
31	92	85	88	88	96.4 / 95.8 / 96.7	-
32	-	-	-	-	-	-
33	-	-	-	-	-	-
34	-	-	-	-	-	-
35	-	402.520	408.150	405.335	-	±3.98

Note: "-" denotes result not provide.

Table 3 (Cont'd) Laboratories' Result for PBDE, BDE-196, BDE-197, BDE-203

Item 2	Sample A : BDE-196					
Lab Code	Result (mg/kg)				Spike Recovery (%)	Expanded Uncertainty (mg/kg)
1	-	-	-	-	-	-
2	-	-	-	-	-	-
3	-	-	-	32.542	-	7.559
4	-	-	-	-	-	-
5	31.8	29.8	-	-	94.9	4.06
6	66.6	-	-	-	-	0.26
7	31.2	-	-	-	88.5 / 104.7 / 91.8	1.59
8	78.51	-	-	-	81.57	0.03
9	-	-	-	-	-	-
10	-	-	-	-	-	-
11	29	30	28	-	-	1.0
12	28.9	29.8	-	29.3	112 / 112	±6.78
13	20.0	21.9	23.2	21.7	93.8 / 96.9 / 97.1 / 95.9	4.62
14	102	94.3	-	-	87.2 / 81.2	6.93
15	26.3	25.5	-	-	83.5 / 87.4	5.1
16	34.5	33.3	-	33.9	87.0	6.78
17	-	-	-	-	-	-
18	50.9	50.7	-	50.8	-	0.164
19	20.0	19.9	19.9	19.9	102	3.49
20	-	-	-	-	-	-
21	19.873	18.980	-	-	85.163	-
22	53.0	51.0	-	52.0	-	±5.2
23	36.7	-	-	-	88	-
24	40.122	-	-	-	118.4	±9.14
25	56.870	57.350	57.120	57.113	89.33 / 90.67 / 90.67	±4.423
26	-	-	-	-	-	-
27	-	-	-	-	-	-
28	38.0	39.2	38.5	38.6	-	±6.7
29	25.4	25.6	31.2	27.4	90	±3.9
30	59.6	56.1	49.7	55.1	-	±8.8
31	25	24	27	25	96.4 / 95.8 / 96.7	±2.05
32	-	-	-	-	-	-
33	-	-	-	-	-	-
34	-	-	-	-	-	-
35	54.649	46.801	57.291	52.913	-	±5.46

Note: "-" denotes result not provide.

Table 3 (Cont'd) Laboratories' Result for PBDE, BDE-196, BDE-197, BDE-203

Item 3	Sample A : BDE-197					
Lab Code	Result (mg/kg)				Spike Recovery (%)	Expanded Uncertainty (mg/kg)
1	-	-	-	-	-	-
2	-	-	-	-	-	-
3	-	-	-	59.063	-	5.059
4	-	-	-	-	-	-
5	98.4	96.2	-	-	94.9	12.6
6	133	-	-	-	-	0.20
7	-	67.8	-	-	88.5 / 104.7 / 91.8	5.21
8	86.61	-	-	-	81.57	0.03
9	-	-	-	-	-	-
10	-	-	-	-	-	-
11	47	46	44	-	-	1.5
12	57.7	58.9	-	58.3	112 / 112	±13.5
13	35.7	35.7	35.7	35.7	93.8 / 96.9 / 97.1 / 95.9	5.25
14	168	155	-	-	87.2 / 81.2	10.7
15	54.0	54.6	-	-	83.5 / 87.4	10.6
16	71.0	79.6	-	75.3	92.0	15.1
17	-	-	-	-	-	-
18	63.9	73.5	-	68.7	-	6.82
19	62.5	62.2	62.4	62.4	102	10.9
20	-	-	-	-	-	-
21	33.094	30.671	-	-	85.163	-
22	93.0	123.4	-	108.0	-	±10.8
23	<1	-	-	-	88	-
24	103.918	-	-	-	118.4	±19.74
25	95.280	92.470	93.950	93.900	89.33 / 90.67 / 90.67	±7.783
26	-	-	-	-	-	-
27	-	-	-	-	-	-
28	72.8	78.8	76.6	76.1	-	±13.2
29	53.2	53.1	55.6	54.0	90	±7.7
30	23.5	16.8	16.7	19.0	-	±3.0
31	54	51	50	52	96.4 / 95.8 / 96.7	±4.99
32	-	-	-	-	-	-
33	-	-	-	-	-	-
34	-	-	-	-	-	-
35	104.744	93.609	95.484	97.930	-	±5.94

Note: "-" denotes result not provide.

Table 3 (Cont'd) Laboratories' Result for PBDE, BDE-196, BDE-197, BDE-203

Item 4	Sample A : BDE-203					
Lab Code	Result (mg/kg)				Spike Recovery (%)	Expanded Uncertainty (mg/kg)
1	-	-	-	-	-	-
2	25.5	24.2	24	24.6	-	±2
3	-	-	-	31.14	-	7.724
4	29.907	29.812	-	29.86		±3.583
5	26.6	24.3	-	-	94.9	3.40
6	42.1	-	-	-	-	0.22
7	-	-	13.8	-	88.5 / 104.7 / 91.8	-
8	75.93	-	-	-	81.57	0.03
9	67.1	70.8	67.9	68.9	-	3.89
10	-	-	-	-	-	-
11	16	16	14	-	-	1.2
12	11.6	12.5	-	12.1	112 / 112	±2.79
13	11.3	11.2	11.9	11.5	93.8 / 96.9 / 97.1 / 95.9	1.82
14	52.1	46.8	-	-	87.2 / 81.2	4.30
15	9.88	9.44	-	-	83.5 / 87.4	1.89
16	26.0	25.9	-	26.0	91.0	5.19
17	-	-	-	-	-	-
18	19.9	18.9	-	19.4	-	0.696
19	40.0	39.8	40.0	39.9	102	7.00
20	-	-	-	-	-	-
21	11.868	11.352	-	-	85.163	-
22	24.7	28.6	-	26.7	-	±2.7
23	105	-	-	-	88	-
24	23.126	-	-	-	118.4	±6.9
25	44.340	44.820	44.050	44.403	89.33 / 90.67 / 90.67	±3.927
26	564.086	551.299	-	557.693	-	±30
27	536.142	528.921	542.163	535.742	82 / 80 / 85	44.352
28	19.3	20.8	20.6	20.2	-	±3.5
29	14.3	13.9	12.1	13.4	90	±2.7
30	31.7	32.9	34.9	33.2	-	±5.3
31	13	10	11	11	96.4 / 95.8 / 96.7	±0.86
32	-	-	-	-	-	-
33	-	-	-	-	-	-
34	-	-	-	-	-	-
35	22.770	21.061	23.871	22.567	-	±1.42

Note: "-" denotes result not provide.

Table 3 (Cont'd) Laboratories' Result for PBDE, BDE-196, BDE-197, BDE-203

Item 5	Sample B : PBDE					
Lab Code	Result (mg/kg)				Spike Recovery (%)	Expanded Uncertainty (mg/kg)
1	-	-	-	-	-	-
2	-	-	-	-	-	-
3	-	-	-	389.237	-	27.900
4	-	-	-	-	-	-
5	217	224	-	-	88.6	-
6	202	-	-	-	-	-
7	-	-	-	-	-	-
8	223.99	-	-	-	81.57	0.03
9	-	-	-	-	-	-
10	-	-	-	-	-	-
11	311	312	309	-	-	1.5
12	273	260	-	267	111 / 115	±61.6
13	71.0	73.4	71.4	71.9	101 / 93.3 / 112 / 102	-
14	327	300	-	-	83.3 / 77.5	20.2
15	281	274	-	-	91.5 / 81.7	51
16	437	481	-	459	-	91.8
17	295	283	280	286	-	62.9
18	292	285	-	289	-	4.90
19	-	-	-	-	-	-
20	-	-	-	-	-	-
21	-	-	-	-	-	-
22	-	-	-	-	-	-
23	608	-	-	-	90	-
24	173.648	-	-	-	118.4	±32.16
25	-	-	-	-	-	-
26	-	-	-	-	-	-
27	-	-	-	-	-	-
28	359	364	369	364	-	±63
29	230	224	264	239	90	±48
30	112.2	123.6	90.3	108.7	-	-
31	83	93	90	88	96.4 / 95.8 / 96.7	-
32	-	-	-	-	-	-
33	-	-	-	-	-	-
34	-	-	-	-	-	-
35	419.750	417.340	-	418.545	-	±1.7

Note: "-" denotes result not provide.

Table 3 (Cont'd) Laboratories' Result for PBDE, BDE-196, BDE-197, BDE-203

Item 6	Sample B : BDE-196					
Lab Code	Result (mg/kg)				Spike Recovery (%)	Expanded Uncertainty (mg/kg)
1	-	-	-	-	-	-
2	-	-	-	-	-	-
3	-	-	-	53.61	-	12.454
4	-	-	-	-	-	-
5	32.3	30.3	-	-	88.6	4.13
6	55.9	-	-	-	-	0.26
7	32.6	-	-	-	88.5 / 104.7 / 91.8	1.59
8	73.04	-	-	-	81.57	0.03
9	-	-	-	-	-	-
10	-	-	-	-	-	-
11	27	27	25	-	-	1.2
12	32.7	31.4	-	32.1	111 / 115	±7.41
13	23.2	24.8	23.8	23.9	101 / 93.3 / 112 / 102	4.50
14	100	92.1	-	-	83.3 / 77.5	6.79
15	29.6	24.3	-	-	91.5 / 81.7	5.3
16	43.0	44.0	-	43.5	83.0	8.70
17	-	-	-	-	-	-
18	38.0	34.4	-	36.2	-	2.52
19	20.0	20.0	20.0	20.0	102	3.50
20	-	-	-	-	-	-
21	18.245	18.417	-	-	67.953	-
22	55.3	40.0	-	47.7	-	±4.8
23	31.2	-	-	-	90	-
24	42.089	-	-	-	118.4	±9.43
25	55.880	56.830	54.920	55.877	84.00 / 85.33 / 86.67	±4.420
26	-	-	-	-	-	-
27	-	-	-	-	-	-
28	37.7	38.4	38.8	38.3	-	±6.6
29	27.5	23.6	31.2	27.4	90	±3.8
30	58.9	62.4	48.4	56.6	-	±9.0
31	23	26	24	24	96.4 / 95.8 / 96.7	±1.97
32	-	-	-	-	-	-
33	-	-	-	-	-	-
34	-	-	-	-	-	-
35	55.056	54.732	40.646	50.146	-	±8.23

Note: "-" denotes result not provide.

Table 3 (Cont'd) Laboratories' Result for PBDE, BDE-196, BDE-197, BDE-203

Item 7	Sample B : BDE-197					
Lab Code	Result (mg/kg)				Spike Recovery (%)	Expanded Uncertainty (mg/kg)
1	-	-	-	-	-	-
2	-	-	-	-	-	-
3	-	-	-	73.224	-	6.271
4	-	-	-	-	-	-
5	102	96.3	-	-	88.6	13.0
6	118	-	-	-	-	0.20
7	-	72.3	-	-	88.5 / 104.7 / 91.8	5.21
8	77.92	-	-	-	81.57	0.03
9	-	-	-	-	-	-
10	-	-	-	-	-	-
11	43	45	43	-	-	1.2
12	63.9	60.3	-	62.1	111 / 115	±14.3
13	35.6	36.4	35.2	35.7	101 / 93.3 / 112 / 102	5.25
14	177	163	-	-	83.3 / 77.5	11.2
15	56.1	57.2	-	-	91.5 / 81.7	11.1
16	88.0	102.0	-	95.0	105	19.0
17	-	-	-	-	-	-
18	55.9	58.1	-	57.0	-	1.55
19	62.5	62.4	62.5	62.5	102	11.0
20	-	-	-	-	-	-
21	31.357	36.913	-	-	67.953	-
22	97.6	111.7	-	105.0	-	±10.5
23	<1	-	-	-	90	-
24	109.014	-	-	-	118.4	±20.63
25	95.960	96.440	96.510	96.303	84.00 / 85.33 / 86.67	±7.785
26	-	-	-	-	-	-
27	-	-	-	-	-	-
28	76.1	75.8	75.4	75.8	-	±13.1
29	52.1	47.7	56.2	52.0	90	±7.3
30	20.9	24.7	12.9	19.5	-	±3.1
31	49	52	53	51	96.4 / 95.8 / 96.7	±4.90
32	-	-	-	-	-	-
33	-	-	-	-	-	-
34	-	-	-	-	-	-
35	103.231	107.183	76.509	95.637	-	±16.68

Note: "-" denotes result not provide.

Table 3 (Cont'd) Laboratories' Result for PBDE, BDE-196, BDE-197, BDE-203

Item 8	Sample B : BDE-203					
Lab Code	Result (mg/kg)				Spike Recovery (%)	Expanded Uncertainty (mg/kg)
1	-	-	-	-	-	-
2	22.9	23.2	23.3	23.1	-	±2
3	-	-	-	39.217	-	9.730
4	33.715	37.51	-	35.613	-	±4.273
5	26.1	26.3	-	-	88.6	3.33
6	27.8	-	-	-	-	0.22
7	-	-	14.5	-	88.5 / 104.7 / 91.8	-
8	73.03	-	-	-	81.57	0.03
9	69.2	72.2	66.4	69.3	-	5.80
10	-	-	-	-	-	-
11	15	14	16	-	-	1.0
12	13.2	12.5	-	12.8	111 / 115	±2.96
13	12.2	12.2	12.4	12.3	101 / 93.3 / 112 / 102	1.95
14	50.2	44.5	-	-	83.3 / 77.5	4.17
15	10.8	8.81	-	-	91.5 / 81.7	1.92
16	29.30	31.00	-	30.2	85.0	6.03
17	-	-	-	-	-	-
18	16.3	14.3	-	15.3	-	1.41
19	40.0	39.9	40.0	40.0	102	7.01
20	-	-	-	-	-	-
21	10.923	11.038	-	-	67.953	-
22	29.2	25.2	-	27.2	-	±2.7
23	106	-	-	-	90	-
24	22.545	-	-	-	118.4	6.9
25	43.140	42.650	42.020	42.603	84.00 / 85.33 / 86.67	±3.925
26	349.361	333.575	-	341.468	-	±30
27	980.468	995.347	972.347	982.72067	84 / 87 / 81	60.034
28	19.0	19.8	20.1	19.6	-	±3.4
29	14.8	12.6	13.0	13.5	90	±2.7
30	32.4	36.5	29.0	32.6	-	±5.2
31	11	15	13	13	96.4 / 95.8 / 96.7	±1.01
32	-	-	-	-	-	-
33	-	-	-	-	-	-
34	-	-	-	-	-	-
35	22.940	22.805	16.736	20.830	-	±3.54

Note: "-" denotes result not provide.

Table 4 Z-Scores of Laboratories

Item 1& Item 5	PBDE			
	Sample A	Sample B	AB-SUM	AB-DIFF
Lab Code	Z-Score	Z-Score	ZB-Score	ZW-Score
1	-	-	-	-
2	-	-	-	-
3	0.7	1.2	1.0	2.7
4	-	-	-	-
5	-0.7	-0.6	-0.7	0.0
6	-0.3	-0.8	-0.6	1.7
7	-	-	-	-
8	-0.3	-0.6	-0.5	0.2
9	-	-	-	-
10	-	-	-	-
11	0.6	0.3	0.5	0.1
12	-0.2	-0.1	-0.2	0.1
13	-2.3	-2.2	-2.2	-0.8
14	0.4	0.4	0.4	-0.6
15	0.0	0.0	0.0	-0.4
16	1.2	1.9	1.6	4.4 ※
17	0.3	0.1	0.2	-0.4
18	0.9	0.1	0.5	3.3 ※
19	-	-	-	-
20	-	-	-	-
21	-	-	-	-
22	-	-	-	-
23	4.2※	3.5※	3.8※	1.4
24	-1.2	-1.1	-1.1	-0.5
25	-	-	-	-
26	-	-	-	-
27	-	-	-	-
28	1.1	0.9	1.0	-0.5
29	-0.3	-0.4	-0.4	-0.6
30	-1.8	-1.8	-1.8	-0.8
31	-2.0	-2.0	-2.0	-1.0
32	-	-	-	-
33	-	-	-	-
34	-	-	-	-
35	1.5	1.5	1.5	0.0

Sample Name	Sample A	Sample B
No. of result	19	19
Median	270	278
Normalized IQR	88.956	94.886
Robust CV (%)	32.9	34.1
Maximum	642	608
Minimum	68.9	71.9
Range	573.1	536.1

Sample Name	AB-SUM	AB-DIFF
No. of result	19	19
Median	387.5	9.9
Normalized IQR	129.996	10.221
Robust CV (%)	33.5	103.3
Maximum	883.9	55.2
Minimum	99.6	0.0
Range	784.3	55.2

| Z—Score | ≥3 are marked by “※” Note:” - “ not applicable

Table 4 (Cont'd) Z-Scores of Laboratories

Item 2 & Item 6	BDE – 196			
	Sample A	Sample B	AB-SUM	AB-DIFF
Lab Code	Z-Score	Z-Score	ZB-Score	ZW-Score
1	-	-	-	-
2	-	-	-	-
3	-0.1	1.0	0.3	6.7※
4	-	-	-	-
5	-0.2	-0.3	-0.4	-0.6
6	1.8	1.1	1.3	3.1 ※
7	-0.2	-0.2	-0.4	-0.2
8	2.5	2.0	2.2	1.2
9	-	-	-	-
10	-	-	-	-
11	-0.3	-0.5	-0.6	0.2
12	-0.3	-0.2	-0.5	0.2
13	-0.7	-0.7	-0.9	0.0
14	3.6※	3.3※	3.4※	0.0
15	-0.4	-0.5	-0.7	-0.4
16	0.0	0.4	0.0	2.7
17	-	-	-	-
18	0.9	0.0	0.3	4.4※
19	-0.8	-0.9	-1.1	-0.7
20	-	-	-	-
21	-0.8	-1.0	-1.1	-0.4
22	1.0	0.6	0.7	0.8
23	0.2	-0.3	-0.3	1.2
24	0.3	0.3	0.2	0.0
25	1.3	1.1	1.1	-0.3
26	-	-	-	-
27	-	-	-	-
28	0.3	0.1	0.0	-0.6
29	-0.4	-0.5	-0.6	-0.7
30	1.2	1.1	1.0	-0.2
31	-0.5	-0.7	-0.8	-0.4
32	-	-	-	-
33	-	-	-	-
34	-	-	-	-
35	1.1	0.8	0.8	0.2

Sample Name	Sample A	Sample B
No. of result	23	23
Median	33.9	36.2
Normalized IQR	17.977	18.273
Robust CV (%)	53	50.5
Maximum	98.2	96.1
Minimum	19.4	18.3
Range	78.8	77.8

Sample Name	AB-SUM	AB-DIFF
No. of result	23	23
Median	54.4	1.5
Normalized IQR	24.269	1.992
Robust CV (%)	44.6	134.1
Maximum	137.4	14.9
Minimum	26.7	0.0
Range	110.7	14.9

| Z – Score | ≥ 3 are marked by “※” Note:” - “ not applicable

Table 4 (Cont'd) Z-Scores of Laboratories

Item 3 & Item 7	BDE-197			
	Sample A	Sample B	AB-SUM	AB-DIFF
Lab Code	Z-Score	Z-Score	ZB-Score	ZW-Score
1	-	-	-	-
2	-	-	-	-
3	-0.3	0.0	-0.1	3.0※
4	-	-	-	
5	0.9	0.8	1.0	-0.1
6	2.1	1.4	1.8	3.2※
7	0.0	0.0	0.1	0.5
8	0.6	0.2	0.4	1.6
9	-	-	-	
10	-	-	-	
11	-0.7	-0.9	-0.7	-0.1
12	-0.3	-0.3	-0.2	0.4
13	-1.0	-1.2	-1.0	-0.6
14	3.0※	3.1※	3.1※	1.4
15	-0.4	-0.5	-0.4	0.0
16	0.2	0.7	0.5	4.4※
17	-	-	-	
18	0.0	-0.5	-0.2	2.4
19	-0.2	-0.3	-0.2	-0.6
20	-	-	-	
21	-1.2	-1.2	-1.1	-0.1
22	1.3	1.0	1.2	0.2
23	-	-	-	
24	1.1	1.1	1.2	0.7
25	0.8	0.7	0.9	0.0
26	-	-	-	
27	-	-	-	
28	0.2	0.1	0.2	-0.5
29	-0.5	-0.7	-0.5	-0.1
30	-1.6	-1.7	-1.5	-0.5
31	-0.5	-0.7	-0.5	-0.4
32	-	-	-	
33	-	-	-	
34	-	-	-	
35	0.9	0.7	0.9	0.0

Sample Name	Sample A	Sample B
No. of result	22	22
Median	68.3	72.8
Normalized IQR	31.413	31.839
Robust CV (%)	46.0	43.8
Maximum	162.0	170.0
Minimum	19.0	19.5
Range	143	150.5

Sample Name	AB-SUM	AB-DIFF
No. of result	22	22
Median	96.3	1.7
Normalized IQR	44.778	2.791
Robust CV (%)	46.5	164.5
Maximum	234.8	13.9
Minimum	27.2	0.0
Range	207.5	13.9

| Z-Score | ≥ 3 are marked by “※” Note: - “ not applicable

Table 4 (Cont'd) Z-Scores of Laboratories

Item 4 & Item 8	BDE-203			
	Sample A	Sample B	AB-SUM	AB-DIFF
Lab Code	Z-Score	Z-Score	ZB-Score	ZW-Score
1	-	-	-	-
2	-0.1	-0.2	-0.1	0.3
3	0.3	0.7	0.5	3.7※
4	0.2	0.5	0.3	2.5
5	0.0	0.0	0.0	-0.1
6	0.8	0.1	0.4	7.0※
7	-0.6	-0.6	-0.6	-0.1
8	2.4	2.4	2.5	1.0
9	2.1	2.2	2.2	-0.3
10	-	-	-	-
11	-0.5	-0.6	-0.6	-0.3
12	-0.7	-0.7	-0.7	-0.1
13	-0.7	-0.8	-0.8	-0.1
14	1.2	1.1	1.1	0.6
15	-0.8	-0.9	-0.9	-0.4
16	0.0	0.2	0.1	1.7
17	-	-	-	-
18	-0.3	-0.6	-0.5	1.7
19	0.7	0.7	0.7	-0.4
20	-	-	-	-
21	-0.7	-0.8	-0.8	-0.2
22	0.0	0.0	0.0	-0.2
23	3.9※	4.2※	4.1※	0.1
24	-0.1	-0.2	-0.2	-0.2
25	0.9	0.8	0.9	0.5
26	25.9※	16.4※	22.0※	112.1※
27	24.8※	50.0※	38.1※	231.5※
28	-0.3	-0.4	-0.3	-0.2
29	-0.6	-0.7	-0.7	-0.4
30	0.4	0.3	0.3	-0.2
31	-0.7	-0.7	-0.7	0.6
32	-	-	-	-
33	-	-	-	-
34	-	-	-	-
35	-0.2	-0.3	-0.2	0.5

Sample Name	Sample A	Sample B
No. of result	28	28
Median	25.75	26.7
Normalized IQR	20.571	19.107
Robust CV (%)	79.9	71.6
Maximum	558	983
Minimum	9.7	9.8
Range	548.3	973.2

Sample Name	AB-SUM	AB-DIFF
No. of result	28	28
Median	37.3	0.6
Normalized IQR	27.192	1.363
Robust CV (%)	72.8	214.2
Maximum	1074.1	316.1
Minimum	13.8	0.1
Range	1060.3	316.0

| Z-Score | ≥ 3 are marked by “※” Note: - “ not applicable

Figure 1 Histogram of Z-Score

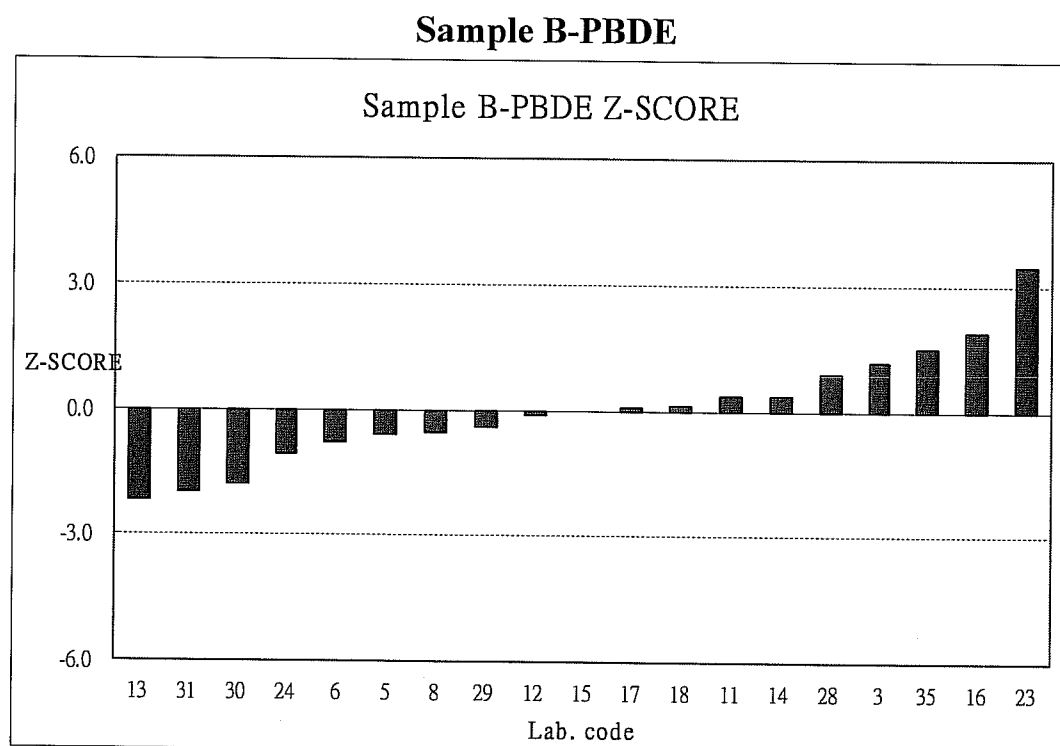
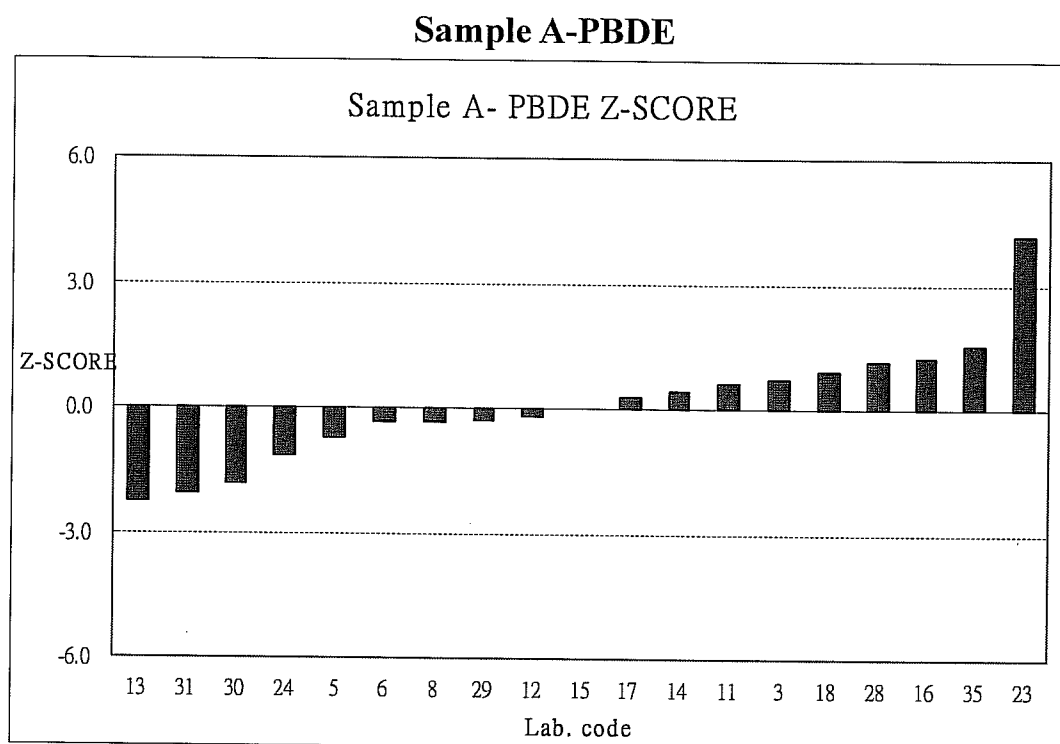
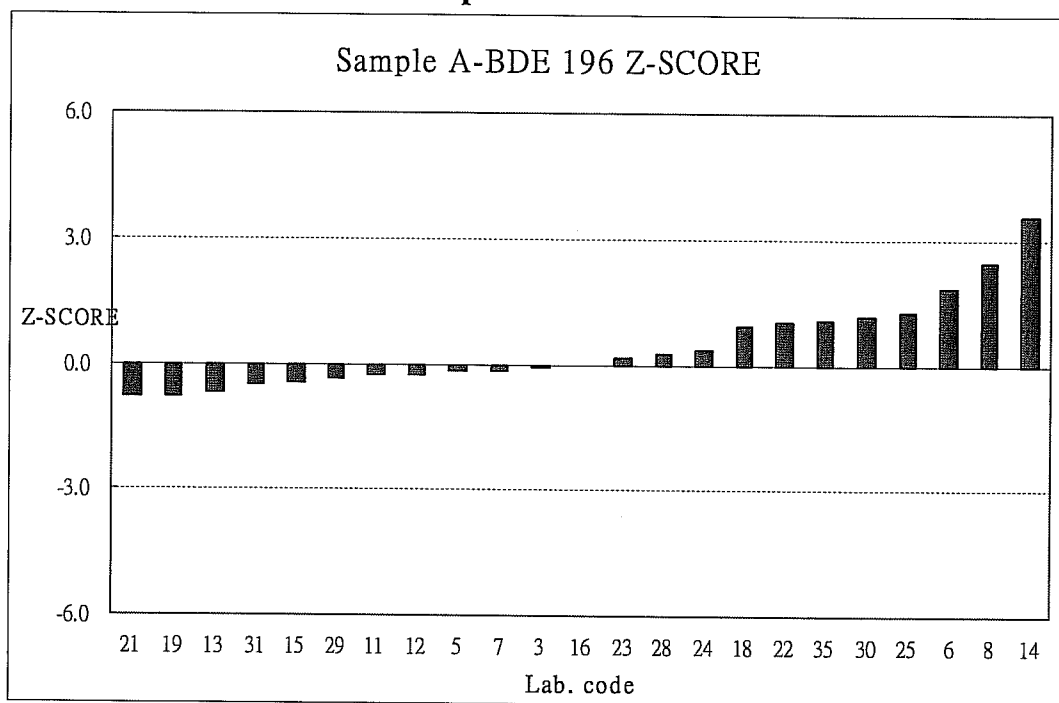


Figure 1 (Cont'd) Histogram of Z-Score

Sample A-BDE 196



Sample B-BDE 196

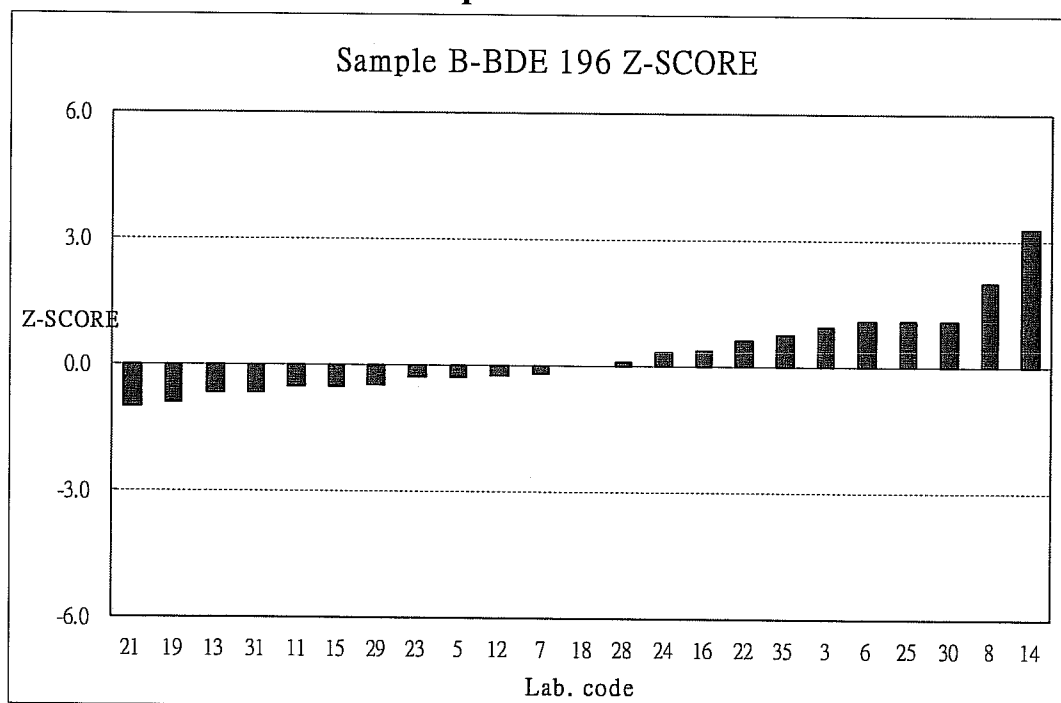
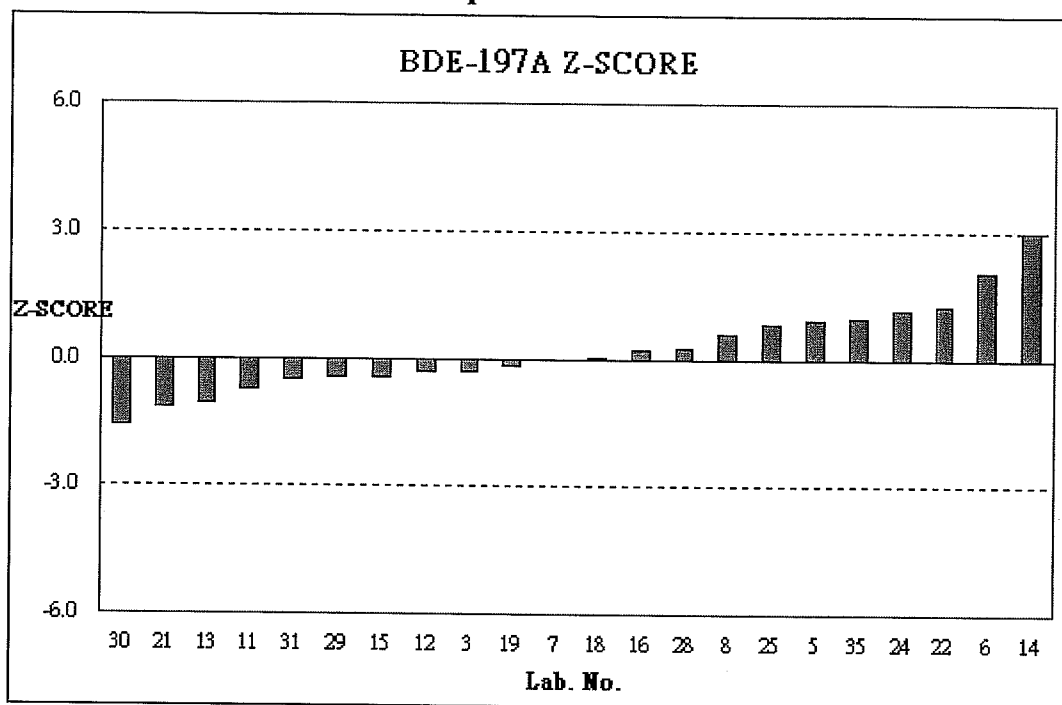


Figure 1 (Cont'd) Histogram of Z-Score

Sample A-BDE 197



Sample B-BDE 197

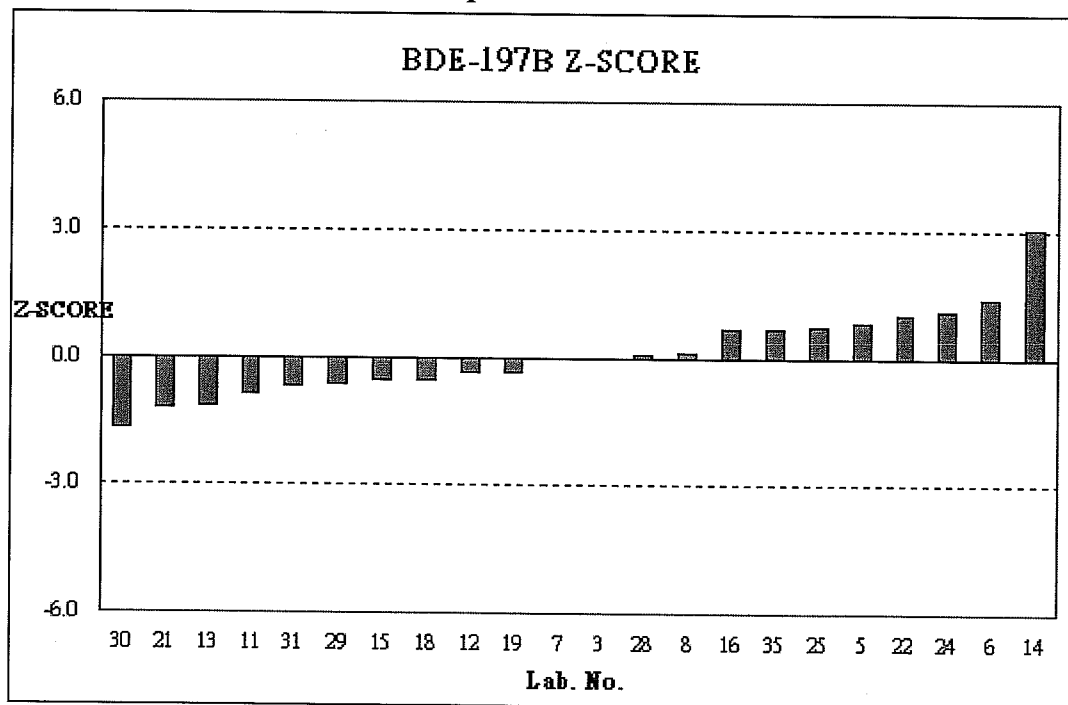
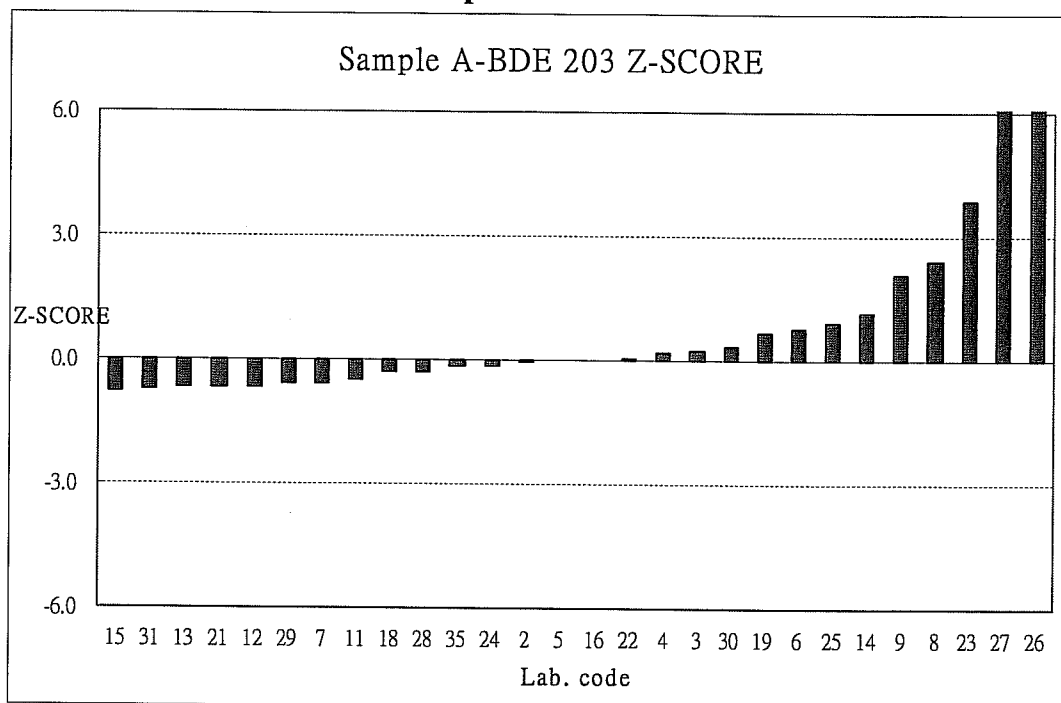


Figure 1 (Cont'd) Histogram of Z-Score

Sample A-BDE 203



Sample B-BDE 203

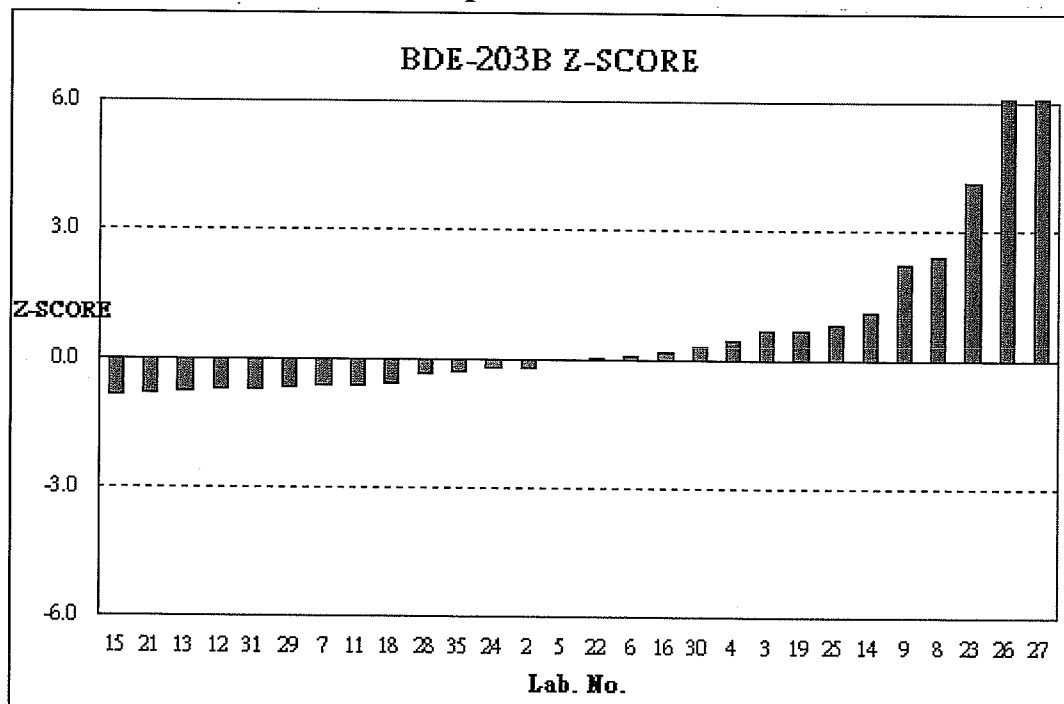
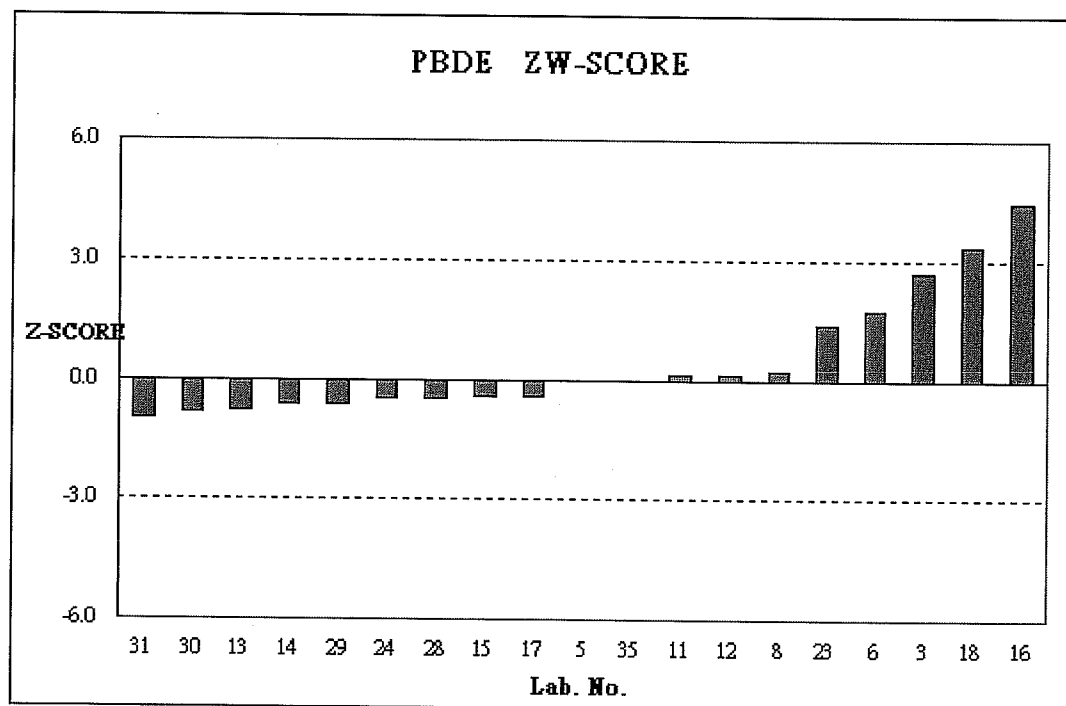
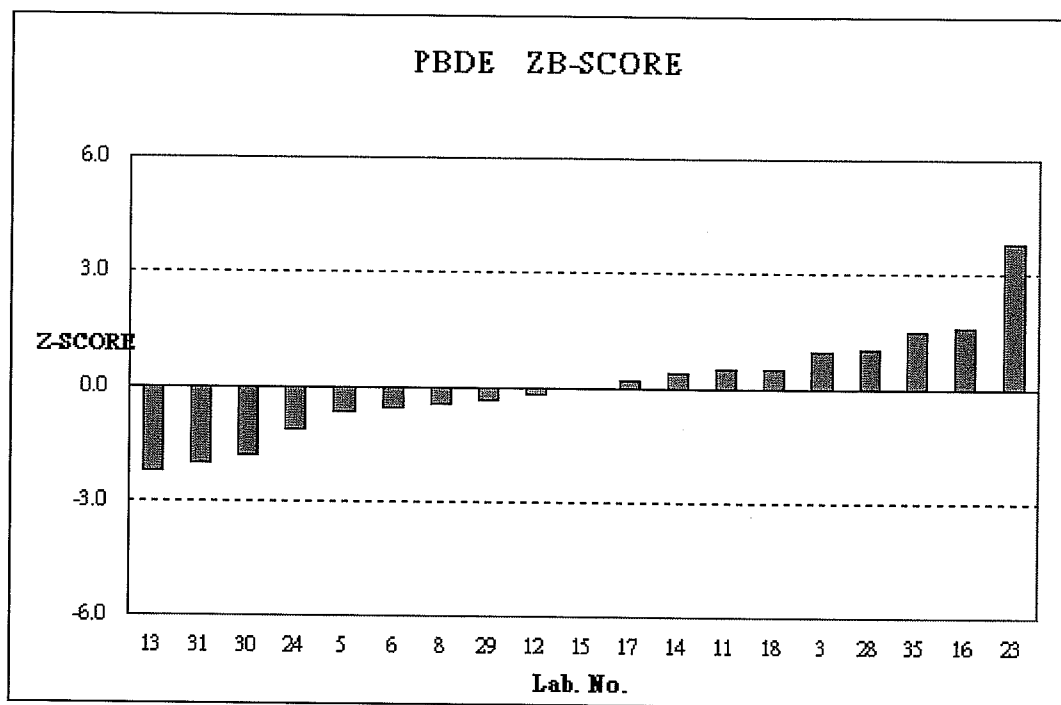


Figure 1 (Cont'd) Histogram of Z-Score

PBDE



BDE-196

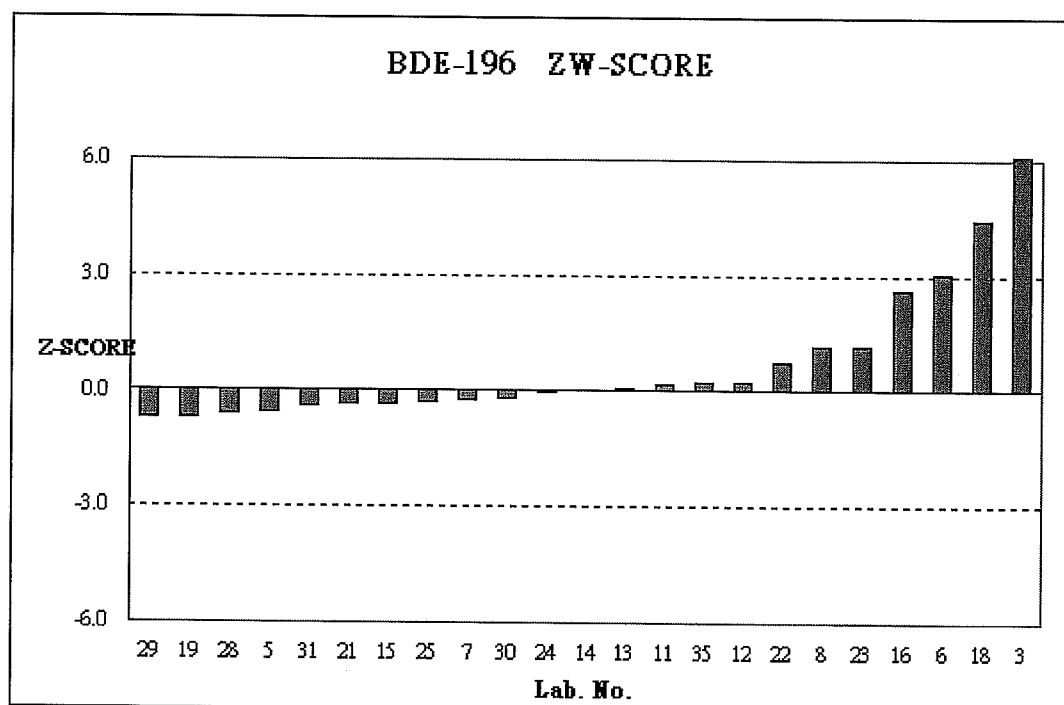
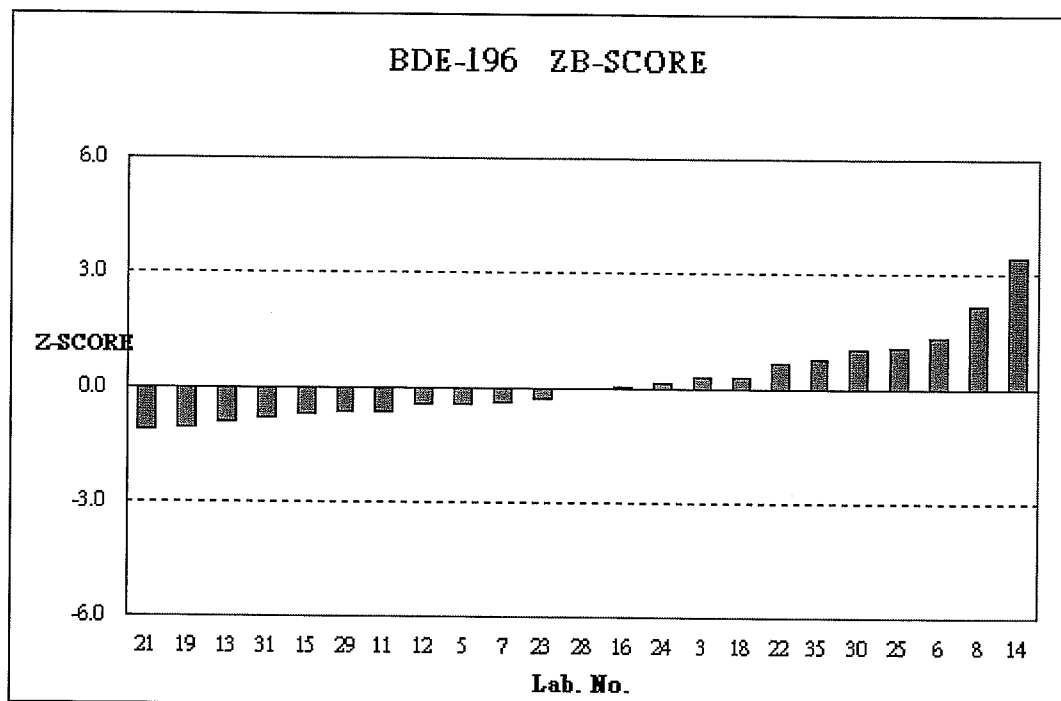
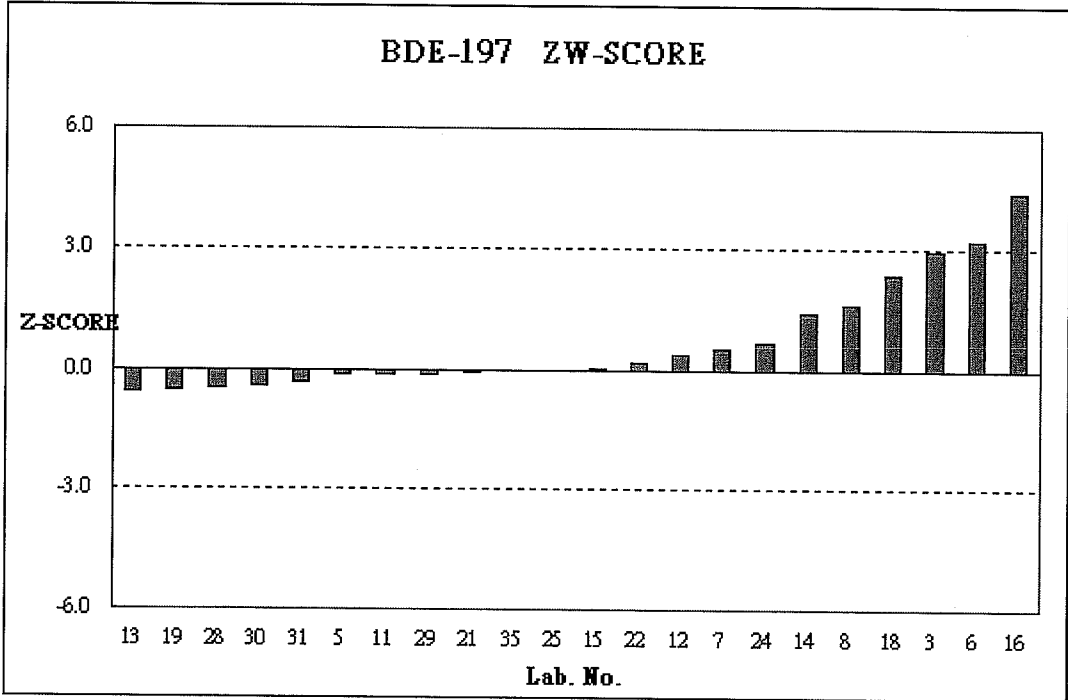
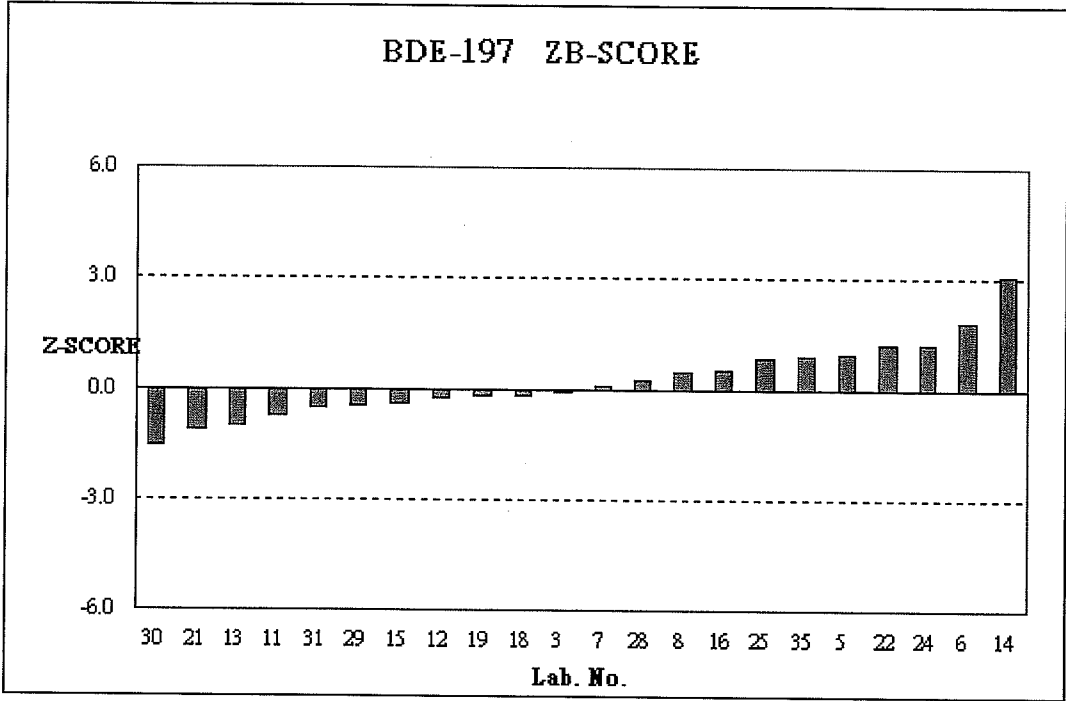


Figure 1 (Cont'd) Histogram of Z-Score

BDE-197



BDE-203

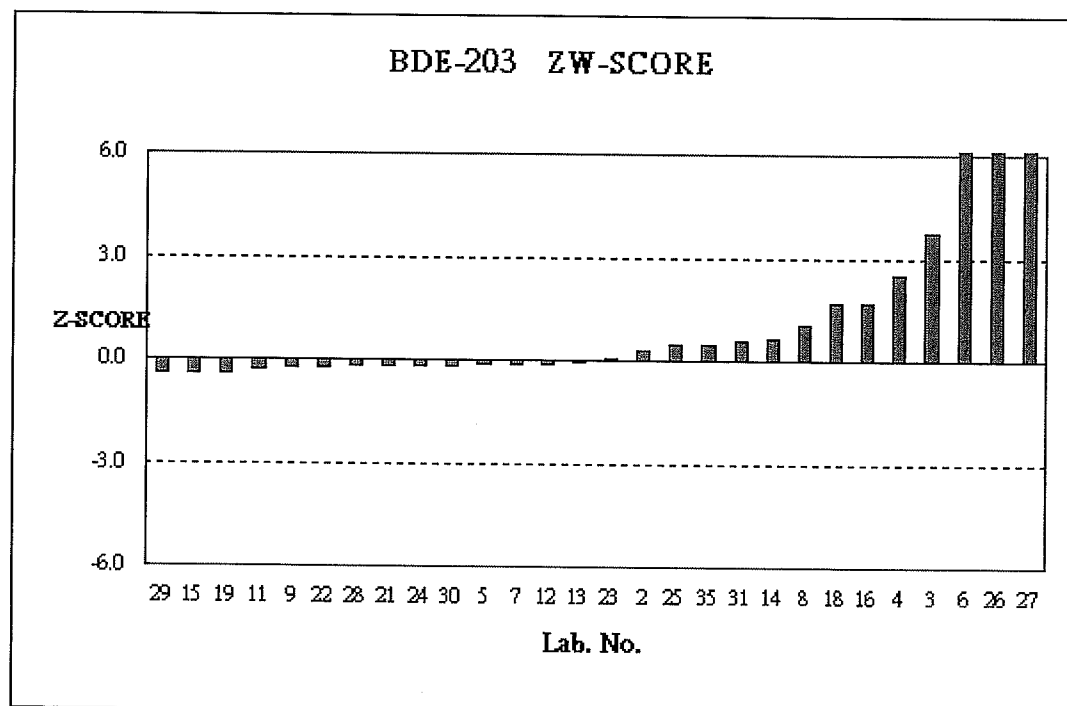
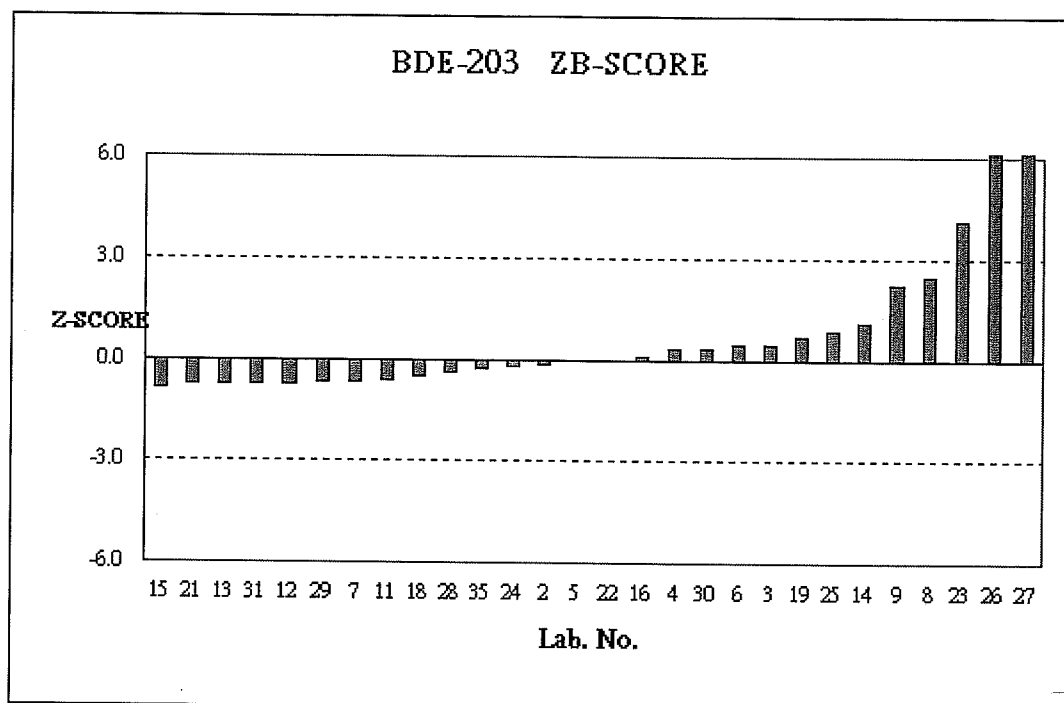


Table 5 Information of Test

Lab Code	Analytical Instrument(s)	Extraction solvent(s)	Extraction technique	Method accreditation
1	-	-	-	-
2	GC/MS: Brand: Pelkin Elmor. GC column: 15m length, internal diameter 0.25µm; film thickness 0.1µm. Temperature program: 100°C hold for 1 min; 340°C hold for 2.00 min, at 15.0°C/min.	Toluene	IEC 62321	YES
3	GC/MS: Brand: Agilent GC F890A/5974C MSD. Column: DB-XLB ; Injection: 2-ul splitless @ 300°C ; Carrier: He ; oven initial Temp: 150°C , ramp@15°C/min to 200°C, ramp@20°C/min to 320°C, hold 15 min.	Toluene	Soxhlet Extraction	YES
4	GC/MS: Brand: Agilent Technologies Column: DB-XLB	Dichloromethane* n-Hexane	Solvent Dissolution	YES
5	GC/MS: Brand: Agilent 6890/5975MS. DB-5MS Column, Film thickness: 0.1µm, Column ID: 0.25mm, length: 15m, 90°C hold 1 min, 20°C/min to 340°C, hold time: 2 min, Injector temp: 280°C	Toluene	Soxhlet extraction for 4 hours	YES
6	GC/MS: Brand: Thermo: TRACE GC Ultra. Therom: DSQ II	Toluene	Soxhlet extraction	
7	GC/MS: Brand: Agilent 6890N/5973N ; colume No : DB-5HT. 100°C for 4 min; 100-200°C at 40°C/min; 200-260°C at 10°C/min; 260-315°C at 30°C/min.	Toluene	Soxhlet extraction	YES
8	GC/MS: Brand: Perkin Elmer/Clarus 500 ; Length: 30 meters ; Agilent J & W GC colume DB-SHT ID: 0.250mm ; Film:0.1µm. Experiment time: 20 min ; injection volume: 1.0 µL ; Initial temperature: 80 deg for 2.00 min ; Ramp1: 20.0 deg/min to 300 deg, hold 5.50 min.	Toluene	IEC 62321 Procedures for the Determination of Levels of six Regulated Substances (Lead, Mercury, Cadmium, Hexavalent chromium, Polybrominated Biphenyls, Polybrominated, Diphenyl Ethers) in Electro technical Products.	YES

Lab Code	Analytical Instrument(s)	Extraction solvent(s)	Extraction technique	Method accreditation
9	GC/MS: Brand: SHIMADZU GCMS-QP2010	Tetrahydrofuran (THF)	Solvent extraction method	NO
10	-	-	-	-
11	GC/MS: Brand: Agilent 6890N/5973i, Agilent technologies	Extraction by acetone	Extraction by ultrasonic wave	YES
12	GC/MS: Brand: Perkin Elmer Clarus 500GCMS. Column: Agilent HP-5MS, 0.25mm*15m*0.1µm. Oven: 80°C for 2 min, 25°C/min ramp to 210°C ; 10°C/min ramp to 310°C for 2 min. Carrier: helium, 1.6mL/min, constant flow. Injector: Splitless, 320°C. Transfer line: 300°C. Ion source temperature: 260°C. Ionization method: electron ionization(EI), 70eV.	Toluene	IEC 62321 Annex A, A.11(dissolution method)	YES
13	GC/MS: Brand: JMS-Q1000GC: JEOL Column: VF-5ms 15M*0.25MM	Toluene /Hexane	Soxhlet Solvent extraction	YES
14	GC/MS: Brand: 6890N/5975(Agilent Technologies Inc.). Column: DB-5HT, 15m(L), 0.25mm(ID), 0.1mm(T). Temp: oven: 100°C *2min, 15°C/min, 320°C *12min. Inject : 270°C. Detector(ion source): 300°C.	Tetrahydrofuran (THF)	Ultra-sonic Extraction	YES
15	GC/MS: Brand: Micromass Autospec Ultima	Tetrahydrofuran (THF)	Ultrasonic Extraction	YES JAB (The Japan Accreditation Board for Conformity Assessment) ISO/IEC 17025 RTL, IEC 62321 Ed.1 Annex. A
16	GC/MS: Brand: Agilent 5975 GC/MSD. Column: DB-5HT.	Toluene	Soxhlet Extraction	NO
17	GC/MS: Brand: Agilent 5975. DB-5HT(15m*0.25mm*0.1µm).	Toluene	Soxhlet	NO

Lab Code	Analytical Instrument(s)	Extraction solvent(s)	Extraction technique	Method accreditation
18	GC/MS: Brand: Agilent Technology Ltd. 7890A, 5975C.	Toluene	Soxhlet Extraction	NO
19	GC/MS: Brand: Agilent, DB-5HT.	Toluene	Soxhlet	NO
20	-	-	-	-
21	GC/MS: Brand: Perkin Elmer Clarus 500.	Toluene	Microwave Extraction	YES
22	GC/MS: Brand: Perkin Elmer Clarus 500. Column: DB-5MS 15m; ID: 0.25mm; 0.1µm.	Toluene	Soxhlet Extraction	YES IEC 62321 Ed1:2008
23	GC/MS: Brand: Perkin Elmer Clarus 500.	Acetone: n-Hexane(1:1)	EPA 3546 & 8270d	YES
24	GC/MS: Brand: Agilent 7890A/5975C. Column: HP-5HT, Length 15m, ID 0.25mm, Thickness 0.1µm. Oven: 100°C for 2 min, 40°C/min ramp to 200°C, 10°C/min ramp to 260°C, 20°C/min ramp to 340°C for 2min.	Toluene	Soxhlet Extraction	NO
25	GC/MS: Brand: Agilent. DB-SMS 0.7µm.	Toluene	Soxhlet Extraction	YES
26	GC/MS: Brand: Perkin Elmer (Clarus 500).	Toluene	Soxhlet Extraction	NO
27	GC/MS: Clarus 500. Brand: Brand: Perkin Elmer. Column: ValcoBond VB-10. Inlet Temp.: 320 °C. Oven Temp.: 70-300 °C. Detector Temp.: 280 °C	Toluene	Soxhlet Extraction	NO
28	GC/MS: Brand: PerkinElmer Clarus 500 GC/MS Column condition: Model PE-1HT, Length 15m, I.D. 0.25mm, Film 0.1µm. Operation condition: GC condition: Inject Temp:320 °C; Flow:1.2 ml/min; Injection volume: 1µL, Splitless Injection. Oven temp program: 1th: Rate(/), Temp 80°C, Hold Time 1min. 2th: Rate 20 °C/min, Temp 320 °C, Hold Time 3min. MS condition: Inlet line temp: 330 °C; Source temp: 270 °C.	Toluene	Microwave Extraction	YES

Lab Code	Analytical Instrument(s)	Extraction solvent(s)	Extraction technique	Method accreditation
29	GC/MS: Brand: Agilent 6890N/5975C. ZB-5HT 15m*0.25mm*0.10µm, splitless He. 1.0ml/min, Oven 120°C (1min) - 25°C/min - 320°C (7min), Inlet Temp 280°C, Transmit line Temp 280°C, EI(+) 250°C, SIM hexaBDE(482,484,643), HeptaBDE(562,564,722), Octa(639,642,801), NonaBDE(719,721,881).	Toluene	Soxhlet Extraction. Others: samples pulverized in liq. Nitrogen; 1.0g sample was added in 30ml Toluene, Soxhlet Extraction for 4hr, then made up to 25ml with toluene.	YES
30	GC/MS: Brand: Agilent GC-MSD.	Toluene	Ultrasonic extraction	YES
31	GC/MS: Brand: Agilent 5975i	Toluene	Soxhlet Extraction 12hours	YES
32	-	-	-	-
33	-	-	-	-
34	-	-	-	-
35	GC/MS: Brand: Agilent 6890*5975 MS decter, Alltech AT-1ht 15m*0.25m*0.1µm, program 110°C hold 4 min ramp@10°C/min to 325°C.	1:1 Toluene Tetrahydrofuran	From IEC 7.3 Sonication	YES

Appendix B

Preparation of the samples

Homogeneity testing

SAMPLE PREPARATION

The sample batch is manufactured by Plastics Industry Development Center. The dosage of brominated flame retardant is set to 0.1%. Technical grade octa-brominated biphenyl ether (the code number: DE-79) is compounded with ABS matrix resin by twin-screw extruder. Specimen is cut with pelletizing machine.

HOMOGENEITY TESTING

The sample batch pellets are loaded separately into 70 bottles. According random number table (table 1) in random sampling way, 15 bottles of test sample are chosen for homogeneity test.

Table 1. Random number table

NOTE 1—Caution: See Note 3>Note 3.																																							
46	96	85	77	27	92	86	26	45	21	89	81	71	42	64	64	58	22	75	81	74	91	48	46	18															
44	19	15	32	63	55	87	77	33	29	45	00	31	34	84	05	72	90	44	27	78	22	07	62	17															
34	39	80	62	24	33	81	67	28	11	34	79	26	35	34	23	09	94	00	60	55	31	63	27	91															
74	97	80	30	65	07	71	30	01	84	47	45	89	70	74	13	04	90	51	27	61	34	63	87	44															
22	14	61	60	86	38	33	71	13	33	72	08	16	13	50	56	48	51	29	48	30	93	45	66	29															
40	03	96	40	03	47	24	60	09	21	21	18	00	05	86	52	86	40	73	73	57	68	36	33	91															
52	33	76	44	56	15	47	75	78	73	78	19	87	06	98	47	48	02	62	03	42	05	32	55	02															
37	59	20	40	93	17	82	24	19	90	80	87	32	74	59	84	24	48	79	17	23	75	83	42	00															
11	02	55	57	48	84	74	36	22	67	19	20	15	92	53	37	13	75	54	89	56	73	23	39	07															
10	33	79	26	34	54	71	35	89	74	08	49	23	17	49	18	81	05	52	85	70	05	73	11	17															
67	59	28	25	47	89	11	65	65	20	42	23	96	41	64	20	30	89	87	64	37	93	36	96	35															
93	50	75	20	09	18	54	34	68	02	54	87	23	05	43	36	98	29	97	93	87	08	30	92	98															
24	43	23	72	80	64	34	27	23	46	15	36	10	63	21	59	69	76	02	62	31	62	47	60	34															
39	91	63	18	36	27	10	78	88	84	42	32	00	97	92	00	04	94	50	05	75	82	70	80	35															
74	62	19	67	54	16	28	92	33	69	98	06	74	35	72	11	68	25	09	95	31	79	11	79	54															

1. Test Procedure

(1) Sample Extraction

- Weigh 100mg grounded test sample and transfer into the extraction thimbles. Record the mass to the nearest 0.1mg.
- Toluene was used as the extraction solvent. Add approximately 60mL extraction solvent in the 100mL round-bottomed flask. The thimble is closed with glass wool to prevent the sample from floating. The equipment is covered with aluminum foil to exclude light and the sample is extracted for at least 2h with each cycle being approximately 2min to 3min. The extract is placed in a 100mL volumetric flask and the round-bottomed flask is rinsed with approximately 5mL of solvent. The

volumetric flask is filled with 100mL solvent.

(2) Calibration

- A. Prepare BDE-196, BDE-197 and BDE-203 stock solution. (1000 μ g/mL)
- B. The following calibration solutions are produced from the stock solution. The different volumes 0.5mL, 1mL, 2mL, 5mL and 10mL are placed in a 100mL volumetric flask separately with a pipette and filled with solvent up to the mark.

(3) Sample Analysis

- A. Analyze the calibration solutions with GC-MS instrument. Quantification is made on the basis of the measurement of the peak area. A calibration curve is developed for quantitative analysis.
- B. After completing calibration curve, sample analysis is executed.

2. Homogeneity Test of Result

The homogeneity tests of results are accomplished by Bureau of Standards, Metrology and Inspection, MOEA, Taiwan. The homogeneity examination of analysis result uses the one-way analysis of variance (one-way ANOVA) method to calculate the F-test value. According to statistics formula, count the values of statistics F. The F marginal value $F_{\alpha}(v_1, v_2)$ is found from the table of the F distribution critical values. To evaluate sample homogeneity, compare statistics F with F marginal value. If $F < F_{\alpha}(v_1, v_2)$ (α is for significance level, v_1 and v_2 are for degree of freedom), then it indicated no special difference among the samples.

Homogeneity test of sample: In this 15 test samples, three kinds of octa-brominated biphenyl ether congeners BDE-196, BDE-197 and BDE-203 are analyzed separately. Each sample reiterates 2 times under the same experiment condition. The test results are shown as the following tables.

(1) BDE-196

unit : mg/kg

Test time Sample No.	1	2	Mean value
40	39.87	35.19	37.53
03	31.33	40.60	35.97
47	42.60	40.13	41.37
24	37.75	36.11	36.93

Test time Sample No.	1	2	Mean value
60	38.61	42.19	40.40
09	37.30	38.81	38.06
21	40.61	41.89	41.25
18	43.00	41.53	42.27
05	42.19	43.18	42.69
52	40.53	42.12	41.33
36	39.69	46.88	43.29
33	41.05	42.28	41.67
52	40.71	33.63	37.17
44	39.08	34.72	36.90
55	38.06	33.91	35.99

(2) BDE-197

unit : mg/kg

Test time Sample No.	1	2	Mean value
40	95.34	81.73	88.54
03	81.99	103.47	92.73
47	108.25	100.90	104.58
24	88.50	90.07	89.29
60	95.99	99.09	97.54
09	90.14	99.21	94.68
21	105.44	100.07	102.76
18	105.63	96.84	101.24
05	103.73	98.27	101.00
52	91.80	96.02	93.91
36	93.04	97.60	95.32
33	102.83	94.35	98.59
52	87.38	79.48	83.43
44	87.03	85.59	86.31
55	87.93	77.75	82.84

(3) BDE-203

unit : mg/kg

Test time Sample No.	1	2	Mean value
40	26.00	20.79	23.40
03	20.82	23.31	22.07
47	25.36	25.53	25.45
24	25.41	21.33	23.37
60	24.27	25.88	25.08
09	23.10	26.00	24.55
21	25.90	26.96	26.43
18	26.51	28.10	27.31
05	26.13	25.02	25.58
52	23.84	26.57	25.21
36	25.48	28.13	26.81
33	23.55	24.77	24.16
52	23.99	20.92	22.46
44	24.61	19.49	22.05
55	23.82	21.20	22.51

3. F-test result of sample homogeneity

(1) BDE-196

Source of ANOVA	Degree of Freedom	Sum of Squares	Mean Square	F value
Between Sample	14	194.1867	13.8705	1.49
Within Sample	15	139.2987	9.2866	

(2) BDE-197

Source of ANOVA	Degree of Freedom	Sum of Squares	Mean Square	F value
Between Sample	14	1348.5036	96.3217	2.39
Within Sample	15	604.7629	40.3175	

(3) BDE-203

Source of ANOVA	Degree of Freedom	Sum of Squares	Mean Square	F value
Between Sample	14	84.7814	6.0558	1.46
Within Sample	15	62.1865	4.1458	

4. Conclusion

From the test results of BDE-196, BDE-197 and BDE-203, the statistics F of three kinds of octa-brominated biphenyl ether congeners is calculated to 1.49, 2.39 and 1.46 separately. In the table of F distribution critical values, F marginal value $F_{0.05}(14,15)=2.40$ at 95 % significance level ($\alpha=0.05$) and F marginal value $F_{0.01}(14,15)=3.67$ at 99 % significance level ($\alpha=0.01$). In this homogeneity test, each statistics F of octa-brominated biphenyl ether is smaller than the F marginal value, this indicated that no significant difference among the mean values of samples at 95 % and 99 % significance level. It means the octa-brominated biphenyl ether in this sample batch is homogeneous.

Appendix C

Instructions to Accreditation Body

Instructions to Participants

Results Sheet



APLAC T070 Determination of PBDE in Acrylonitrile butadiene styrene (ABS)

Proficiency Testing Program

Instructions to Accreditation Bodies

1. ORGANIZATION

The Proficiency Testing Program on “Determination of PBDE in Acrylonitrile butadiene styrene (ABS)” is organized by Taiwan Accreditation Foundation (TAF) under the auspices of Asia Pacific Laboratory Accreditation Cooperation (APLAC). The main purpose of this program is to evaluate the competence of participating laboratories on the concerned tests.

2. SAMPLES

Accreditation bodies (AB) will receive PT samples and instruction information (hard copy and soft copy) from the organizers. Each laboratory is supplied with two amber bottles that each contains about 7 gram of Acrylonitrile butadiene styrene (ABS) pellets. Upon receipt, AB should carefully check the samples (number of bottles, physical conditions, etc.) and promptly acknowledge the TAF coordinator by returning the Receipt Form (for Accreditation Bodies) through E-mail and facsimile to

Taiwan Accreditation Foundation (TAF)

Ms. Cherry Cheng

FAX: +886-3-5726308

E-mail: cherry@taftw.org.tw

3. SAMPLES DISTRIBUTION

AB is recommended to promptly distribute the samples, together with the Instructions to Participating Laboratories and Results Sheet (hard and soft copy) to their respective participating laboratories. The samples shall be stored at room temperature before dispatch.

AB shall remind their participating laboratories to carefully check the samples upon receipt. New samples will be re-sent for any damaged samples.

4. **TESTS TO BE CARRIED OUT**

Please refer to the Instructions to Participating Laboratories enclosed, which outline the tests to be performed and the instructions for test method and reporting results.

The Instructions to Participating Laboratories documents are master copies. The AB should make copies for distribution to the participating laboratories. Instructions could be e-mailed on request. If necessary, these documents should be translated into the appropriate language by the accreditation body.

5. **SAMPLE DISTRIBUTION**

Each accreditation body shall be responsible for the samples dispatching to the respective participating laboratories within one week of receipt date and the delivery costs in their own economy.

6. **DOCUMENTS TO BE SUBMITTED**

Within one week of the completion of the testing, participating laboratories are required to both facsimile and e-mail to their accreditation body the Results Sheet requested in the Instructions to Participating Laboratories. No other documentation is required. The accreditation body should ensure that the information has been supplied by the participating laboratories and is complete. No later than **August 15, 2009**, AB is required to forward these complete results forms to the TAF coordinator yuanchen@taftw.org.tw by **E-mail/Facsimile**.

7. **GENERAL INFORMATION**

For general queries, please contact:

Taiwan Accreditation Foundation (TAF)

Ms. Cynthia Chen

E-mail:yuanchen@taftw.org.tw

Ms. Cherry Cheng

FAX: +886-3-5726308

E-mail:cherry@taftw.org.tw



APLAC T070 Determination of PBDE in Acrylonitrile butadiene styrene (ABS)

Proficiency Testing Program

Receipt Form

In order to monitor the progress of the Proficiency Testing Program, we kindly ask each accreditation body, on receipt of the samples, to fill in this **Receipt Form** and E-mail/facsimile it to:

Taiwan Accreditation Foundation (TAF)

Ms. Cherry Cheng

FAX: +886-3-5726308

E-mail: cherry@taftw.org.tw

Thank you in advance for your cooperation.

1. The APLAC T070 samples were received on: _____ (date).
2. After inspection, are the contents of the package damaged:
☐ Yes ☐ No, if yes, is it still suitable for use? ☐ Yes ☐ No
3. Is there a Declaration to Customs Officials and Shipping Agents enclosed in the plastic envelope and attached to the outside of the case? ☐ Yes ☐ No

Remarks: _____

Name of Accreditation Body: _____

Contact Person: _____

Fax: _____

E-mail: _____



APLAC T070 Determination of PBDE in Acrylonitrile butadiene styrene (ABS)

Proficiency Testing Program

Instructions to Participating Laboratories

1. ORGANIZATION

The Proficiency Testing Program on “Determination of PBDE in Acrylonitrile butadiene styrene (ABS)” is organized by Taiwan Accreditation Foundation (TAF) under the auspices of Asia Pacific Laboratory Accreditation Cooperation (APLAC). The main purpose of this proficiency testing program is to evaluate the competence of participating laboratories on the concerned tests.

2. SAMPLES

Participating laboratories will be given two amber sample bottles labeled APLAC A and APLAC B from their respective accreditation bodies. Each bottle contains about 7 gram of ABS pellet. Upon receipt of the samples, participating laboratories should carefully inspect the sample for any physical damages and defects. New samples will be replaced for any damaged claims. Intact sample is recommended to be stored in a secure environment at room temperature before use.

3. TESTS TO BE PERFORMED

Participating laboratories are required to determine the concentration (in mg/kg) of PBDE in the sample. Uncertainties should be calculated using the method in the ISO/IEC Guide 98-3:2008, Uncertainty of measurement -- Part 3: Guide to the expression of uncertainty in measurement.

4. RESULTS AND DOCUMENTS TO BE SUBMITTED

Report all the data in Results Sheet, where possible, estimate and report the expanded uncertainty ($\pm$ mg/kg) for individual analyze. Submit the results directly to Ms. Cynthia Chen (E-mail: yuanchen@taftw.org.tw or Fax No.: +886-3-5726308) before August 15, 2009. Unless with special reasons, results submitted after the deadline might not be accepted by the organizers.

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 (at least until a final report has been issued).

5. CONFIDENTIALITY

Each laboratory will be assigned with a unique identification code. This unique code will be used throughout the programme. All information on the identities and results of participating laboratories will be kept confidential and not be disclosed.

6. GENERAL INFORMATION

For general queries, please contact with your accreditation body, additional information may be obtained from:

Taiwan Accreditation Foundation (TAF)

Ms. Cynthia Chen

E-mail: yuanchen@taftw.org.tw

Ms. Cherry Cheng

E-mail: cherry@taftw.org.tw



APLAC T070 Determination of PBDE in Acrylonitrile butadiene styrene (ABS)

Lab Cod

Results Sheet

Name of Accreditation Body: _____

Name of Participating Laboratory: _____

Contact person/E-mail: _____

Test Date: _____ (day/month/year)

Part I: Test Results

	Test	Result(mg/kg)				Measurement Uncertainty (mg/kg)
Sample A	PBDE					
	BDE-196					
	BDE-197					
	BDE-203					
Spike Recovery (%)						
Sample B	PBDE					
	BDE-196					
	BDE-197					
	BDE-203					
Spike Recovery (%)						

Note: Report all tests to 3 significant figures or 3 decimal places, whichever is appropriate

Part II: Method Information

1. Analytical Instrument(s): GC/MS: Brand: _____

(please specify column and operation condition)

2. Extraction solvent(s): _____

3. Extraction technique: _____

Others

(please specify)

4. Method accreditation: YES; NO_____

5. Other additional information:

* Please circle as appropriate

- **It is the responsibility of the participants to avoid collusion and falsification of result so as to ensure a reliable assessment to be made in this proficiency testing programme**
- Please submit this “Results sheet” E-mail and facsimile to the TAF coordinator
yuanchen@taftw.org.tw

Signature: _____

Date: _____

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 measurand” (the measurand is the particular quantity subject to measurement).

The result of a test or measurement is our best estimate of the true value of the measurand. The result is imperfect. The true value of the measurand 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.

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 recognize 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, inhomogeneity 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 internet⁷.

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 utilizing 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 utilize 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 ($u(x_i)$) is an input component of uncertainty x_i 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 (c_i) 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 $u(x_i)$ for each input component (x_i) with its respective sensitivity coefficient c_i to produce $c_i u(x_i)$ and then combining those values by taking the square root of the sum of their squares. Note that the products $c_i u(x_i)$ 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 measurand at a given level of confidence ($U = k \cdot uc(y)$). 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 17025:2005, General requirements for the competence of testing and calibration laboratories.
2. ISO/IEC Guide 98-3:2008, Uncertainty of measurement -- Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)
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. 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)

I

Appendix D

Statistical Procedures

Calculations and Formulae

STATISTICAL PROCEDURES

The procedures are based on robust statistics and use z-score to assess performance of participant. Z-score are normalized value that gives a score to each result, relative to the other numbers in the group, so a z-score value close to zero means that result agrees well with those from the other laboratories.

Robust statistics are statistics that are not highly influenced by the presence of extreme results. In a mathematical environment robustness is the ability of a statistical method center of a database, the mean (average), is not robust. A robust alternative to the mean is the median (the middle value). The difference between these two is best illustrated by the following example.

The statistical procedures use z-score to identify outlier results. An outlier will be any result(s), which has an absolute z-score value greater than three. Outliers are identified in the table by a marker (✖) next to the relevant z-scores.

The calculation of the z-score depends on the statistical design of the program. Related samples can be identical (uniform pair) or similar (split pair). Occasionally it may be the case that a program can only be designed to have a single result on a single sample.

A very high between-laboratories z-score indicates that one or both of a laboratory's results is significantly higher than the consensus value (median). Similarly, a very low (i.e. negative) between-laboratories z-score shows that a laboratory's result(s) is lower than expected.

Summary of 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 normalized interquartile range of the results (*Normalized IQR*) – the interquartile range times 0.7413;
4. The robust coefficient of variation, expressed as a percentage (*Robust CV*) - i.e.
$$100 \times \text{Normalized IQR} \div \text{Median};$$
5. The minimum and maximum laboratory results; and
6. The range (*Maximum - Minimum*).

Ordered Z-Score Charts

Ordered between laboratories z-score bar charts and ordered within laboratory z-score bar charts are also used to illustrate the data. Each laboratory z-score is drawn on a chart and identified by their code number. The z-score limits of ± 3 are also drawn which makes it easy to identify any bars extending past this point which would be outliers.

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

Suppose we have pairs of results (for related samples) from a number of laboratories. Let A_i be the result for the first sample (A) from laboratory i . Similarly, B_i is the result from laboratory i for the second sample (B). The number of (pairs of) results is denoted by N .

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* which a quarter of the results lie. Similarly, the upper quartile ($Q3$) is the value *above* which a quarter of the results lie. The quartiles are calculated analogously to the median and $IQR = Q3 - Q1$. The "Normalized 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 normalized 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).

Robust z-scores are calculated by replacing the mean and standard deviation in the "classical" z-score by the median and normalized IQR, respectively - i.e. the z-score for a result is the result minus the median (all) divided by the normalized IQR. As indicated above, the between-laboratories and within-laboratory z-scores are based on the (standardized) sum and difference of the pair of results.

Let S_i be the standardised sum of laboratory's results. Also, D_i is the standardised difference between the two results from laboratory i . Now

$$S_i = \frac{(A_i + B_i)}{\sqrt{2}}$$

$$D_i = \frac{(A_i - B_i)}{\sqrt{2}} \quad \text{if } (A_i) > (B_i),$$

or

$$D_i = \frac{(B_i - A_i)}{\sqrt{2}} \quad \text{if } (A_i) < (B_i).$$

So, the between-laboratories z-score for laboratory i (denoted ZB_i) is the robust z-score of its S_i and their within-laboratory z-score (ZW_i) is the robust z-score of its D_i - i.e.

$$ZB_i = \frac{S_i - \text{median}(S_i)}{IQR(S_i) \times 0.7413}$$

and

$$ZW_i = \frac{D_i - \text{median}(D_i)}{IQR(D_i) \times 0.7413}$$