DETERMINATION OF CADMIUM AND LEAD IN HERBAL SAMPLE PROFICIENCY TESTING PROGRAMME

APLAC T065

FINAL REPORT

21 Oct 2008

CONTENTS

Summary of Results

1. The proficiency testing programme (APLAC T065) aimed at evaluating the testing capability of participants on the quantitative analysis of cadmium and lead in a herbal sample, *Herba Desmodii Styracifolii*.

2. A total of 109 laboratories from 42 economies enrolled in the programme and 101 of them returned their results to the organizer.

3. The reference values of cadmium and lead were assigned by the HKGL using a gravimetric isotope dilution inductively coupled plasma-mass spectrometry (ID-ICP-MS) technique. The standard deviations for proficiency assessment were calculated using the Horwitz Equation[8.1]. z-Score is used as the numerical indicator to assess individual participant's competence relative to the others in the programme.

4. z-Scores of participants are summarized as follows:

   (96 valid numeric data from participants were used in the assessment of z-score)

Performance	**Number of Participants (Percentage)**	
	Cadmium	**Lead**
$\|z\| \leq 2$	81 (84.4 %)	75 (78.1 %)
$2 < \|z\| < 3$	8 (8.3 %)	2 (2.1 %)
$\|z\| \geq 3$	7 (7.3 %)	19 (19.8 %)
Total:	96 (100 %)	96 (100 %)

1. Introduction

1.1. Treatments with herbal medicines are commonly advocated for a wide range of conditions in many Asian countries and have recently been become popular in the West. However, the ubiquitous presence of heavy metals in the environment arising from various natural and anthropogenic activities has led to the accumulation intrusion of these substances into agricultural products including herbal plants. The literature information[8.2] revealed that cadmium and lead were mostly found in a wide variety of herbs and their concentrations were respectively in the range of 0.2 to 2.7 mg/kg for cadmium and 0.1 to 2.8 mg/kg for lead.

1.2. A proficiency testing programme (APLAC T065) on the quantitative analysis of cadmium and lead in a herbal sample, *Herba Desmodii Styracifolii* was organised by the Hong Kong Government Laboratory (HKGL), in collaboration with Hong Kong Accreditation Service (HKAS), and under the auspices of the Asia-Pacific Laboratory Accreditation Co-operation (APLAC).

1.3. A total of 109 laboratories from 42 economies enrolled in the programme and 101 of them returned their results on or before the deadline (Table I). Each participant was confidentially assigned with a unique laboratory code (1 to 102) and the code was used throughout the programme.

2. Objectives

2.1. The main objective of this programme is to assess the performance of participants in the analysis of cadmium and lead in herbal sample through interlaboratory comparison.

2.2. Test results from participants are evaluated according to the statistical techniques outlined in ISO13528:2005[8.3] "Statistical methods for use in proficiency testing by interlaboratory comparisons" and in the international harmonized protocol for the proficiency testing of analytical chemistry laboratories[8.4]. z-Score is used as a numerical indicator to assess individual participant's competence relative to the others in the programme.

3. Test Material

3.1. Several batches of dried samples of *Herba Demodii Styracifolii* were purchased from the local market and authenticated by the HKGL. The samples that contained trace quantities of incurred cadmium (ranging of 0.1 to 0.5 mg/kg level) and lead (ranging of 1 to 5 mg/kg level) were rinsed with distilled water to remove dirt and foreign matters. The samples were air-dried in a Clean Room (Class 1000), then subject to freeze drying, grinding, sieving (through 250 μm sieve) and thorough mixing. The collected fine powder was packed into pre-cleaned and nitrogen-flush amber glass bottles, each in 5 g portion. The bottles were disinfected with γ-irradiation at about 1 kGy, and sealed under vacuum inside polypropylene bag. More than 300 bottles of herbal samples was finally prepared and stored at room temperature before shipment.

3.2. Homogeneity (performed in November 2006) and stability tests (at 25ºC from December 2006 to September 2008 and at 37ºC from December 2006 to November 2007) of the samples were determined in accordance with the procedures stipulated in APPENDICES I and II. Results indicate the samples are suitable to be used in the PT programme.

3.3. Samples together with “Instructions to Accreditation Bodies”, “Receipt Form”, “Instructions to Participating Laboratories”, and “Result Proformas” were sent to participating accreditation bodies (AB) in July 2008. A specimen copy of these instructions was given in APPENDICES III to VII.

3.4. All AB were requested to read the “Instructions to Accreditation Bodies” and to redirect the sample(s) and the attached documents to their nominated laboratories as soon as possible.

3.5. Each participant was provided with one bottle of 5 g of test sample in the programme.

4. Test Required

4.1. Participants were required to determine, preferably in triplicate, the concentration of cadmium and lead in the test sample according to their appropriate validated methods.

4.2. Participants were requested to report results in mg/kg to three significant figures or three decimal places, whichever was appropriate.

4.3. Expanded measurement uncertainty and technical information of the method used were required.

4.4. All the analytical results and information should be reported in the Result Proformas provided.

5. Statistical Evaluation

5.1. Participants' test results

5.1.1. Test results received from participants are presented in TABLES II to III. Their methodologies and techniques employed as well as their accreditation status are tabulated in TABLES IV to VI.

5.2. Performance evaluation

5.2.1. Performance of each participant was evaluated by means of a performance index, z-score, which is calculated as:

$$z = \frac{x_i - m}{S}$$

where x_i = reported mean of individual participant
m = computed mean from robust statistics
S = Standard deviation (SD) estimated from Horwitz Equation

5.2.2. z-Score is interpreted as follows:

(a)	$\|z\| \leq 2$	Satisfactory
(b)	$2 < \|z\| < 3$	Questionable
(c)	$\|z\| \geq 3$	Unsatisfactory

5.2.3. Participants having z-score(s) ≥ 3 or ≤ -3 should thoroughly investigate their results and reported the findings to their AB. Participants having z-score(s) in the range $2 < |z| < 3$ are encouraged to review their results.

6. Results and Discussions

6.1. An interim report was prepared by the HKGL and issued to participants and their ABs on 17 September 2008. Participants were requested to check the correctness of their submitted data and to inform the organizers of any mistakes found on or before 29 September 2008. Some laboratories informed the HKGL that they had miscalculated their submitted data and requested to amend accordingly. They were informed that it was their responsibility of participants to ensure the correctness of the submitted results, and therefore no data correction was made in the interim report.

6.2. Number of participants' data submitted is summarized as follows:

	Cadmium	**Lead**
No. of data submitted	101	101
No. of valid numeric data	96	96

6.3. Assigned values

6.3.1. Assigned values of cadmium and lead in the sample were determined by a gravimetric isotope dilution inductively coupled plasma-mass spectrometry (ID-ICP-MS) method. Results of the ID-ICP-MS method were verified in the Comité Consultatif pour la Quantité de Matière (CCQM) inter-laboratory comparison (CCQM P-97) and found to have good degree of equivalence, in terms of accuracy and precision, with other participating national metrology institutes. The assigned values were the average of four independent analyses that performed in different periods.

6.3.2. Results of assigned values of cadmium and lead

	Cadmium	**Lead**
Assigned value (mg/kg)	0.2797	1.5065
Expanded uncertainty (mg/kg) at $k = 2$	0.0067	0.0226

6.4. Horwitz's standard deviation used for z-score assessment

	Cadmium	Lead
Horwitz's SD (%)	19.2	14.9
Horwitz's SD (mg/kg)	0.0538	0.2251

6.5. Distribution of participants' pooled data calculated using robust statistics for cadmium and lead are 18.7% and 19.8% respectively, which are comparable with those of the Horwitz SD. The robust mean values of participants' data (Cd = 0.2787 mg/kg and lead = 1.466 mg/kg) are also in good agreement with the assigned values. The results indicated the overall performance of participants is satisfactory.

6.6. z-Scores of individual participant are presented in TABLE VI and illustrated graphically in HISTOGRAMS I to II. Performance of participants is summarized as follows:

Performance	Number of Participants (Percentage)	
	Cadmium	Lead
$\|z\| \leq 2$	81 (84.4 %)	75 (78.1 %)
$2 < \|z\| < 3$	8 (8.3 %)	2 (2.1 %)
$\|z\| \geq 3$	7 (7.3 %)	19 (19.8 %)
Total:	96 (100 %)	96 (100 %)

In brief, 22 participants (Lab. Code: 2, 11, 21, 23, 27, 28, 35, 38, 43, 44, 45, 49, 50, 62, 63, 71, 74, 77, 90, 98, 99, 102) were identified as having reported one or more unsatisfactory results; and 26 out of the 192 results submitted were identified as unsatisfactory results (13.5 %).

6.7. Majority of participants used ICP-MS (41 laboratories) for their analysis, which followed by GFASS (31); ICP-AES/OES (18) and FAAS (14). Microwave assisted digestion (57 laboratories) was the commonly

used digestion method and the remaining used either wet or dry ashing method. All of them used nitric acid or a combination of nitric acid with HCl, H_2O_2, $HClO_4$ or HF for sample digestion. (TABLE IV)

6.8. Apart from Lab. 45, the recovery of both analytes was satisfactory. Three laboratories used CRM as their recovery quality control. (TABLE V).

7. Remarks

7.1. Efforts and supports from all AB and participants to this programme are gratefully noted. Participants are welcome to forward any comments on this PT to the following address:

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8. References

8.1. W. Horwitz. Evaluation of analytical methods used for regulations of food and drugs, *Anal. Chem.*, **1982**, 54:67A-76A.

8.2. U. Divrikli, N. Horzum, M. Soylak, L. Elci, *Int. J. Food Sci. Technol.*, **2006**, 14:712-716.

8.3. ISO 13528 “Statistical methods for use in proficiency testing by interlaboratory comparisons”, **2005**, Geneva, Switzerland.

8.4. M. Thompson, S.L. Ellison, R. Wood. The International harmonized protocol for the proficiency testing of analytical chemistry laboratories (IUPAC technical report), *Pure Appl. Chem.*, **2006**, 78:146-196.

TABLE I: List of Participating Accreditation Bodies

No.	*ECONOMIES	PARTICIPANTS ENROLLED	RETURNED RESULTS
1	Argentina	2	2
2	Australia	3	2
3	Belgium	2	2
4	Brunei	1	1
5	Canada	4	4
6	China	4	4
7	Costa Rica	1	1
8	Croatia	1	1
9	Czech Republic	1	1
10	Cuba	2	0
11	Ecuador	1	1
12	Estonia	2	2
13	Finland	2	2
14	Germany	2	2
15	Greece	1	1
16	Guatemala	1	1
17	Honduras	2	2
18	Hong Kong	13	13
19	Indonesia	4	1
20	Israel	2	2
21	Jamaica	2	2
22	Japan	4	4
23	Jordan	2	2
24	Korea	4	4
25	Latvia	1	1
26	Malaysia	2	1
27	Malta	1	1
28	Mexico	3	3
29	New Zealand	3	3
30	Norway	1	1
31	Philippines	4	3
32	Poland	4	4
33	Romania	2	2
34	Singapore	4	4
35	Slovenia	1	1
36	Sri Lanka	2	2
37	Sweden	2	2
38	Taiwan	4	4
39	Trinidad & Tobago	1	1
40	UK	2	2
41	USA	5	5
42	Vietnam	4	4
	Total:	**109**	**101**

TABLE II: Participants' Results for Cadmium

Lab. Code	Concentration (mg/kg)				Measurement Uncertainty	
	Data #1	Data #2	Data #3	Mean	Expanded Uncertainty (mg/kg)	Coverage Factor
1	0.310	0.312	0.306	0.31	0.05	2
2	0.273	0.272	0.280	0.275	0.1	95%
3	0.290	0.255	0.268	0.271	0.092	2
4	0.249	0.275	0.260	0.261	0.065	2.776
5	0.16	0.16	0.20	0.17	0.03	2.0
6	0.266	0.246	0.252	0.255	0.026	2
7	0.280	0.279	0.281	0.280	0.054	2
8	0.290	0.280	0.280	0.283	0.019	2
9	0.259	0.249	0.238	0.249	0.0195	6
10	0.27	0.25	0.28	0.267	0.031	2
11	12.3	10.1	10.9	11.1	0.33	1.65
12	0.284	0.296	0.302	0.294	0.0441	2
13	0.388	0.216	0.385	0.330	0.068	2
14	0.37	0.38	0.48	0.41	0.16	2
15	0.281	0.279	0.275	0.278	0.038	2
16	0.226	0.224	0.226	0.225	0.048	2
17	0.278	0.274	0.280	0.277	0.022	2
18	0.206	0.200	0.214	0.207	0.02	2
19	0.263	0.257	0.256	0.259	0.008	2
20	0.245	0.246	0.248	0.246	0.025	2
21	2.769	3.151	3.731	3.22	1	2
22	0.295	0.298	---	0.297	0.035	2
23	0.381	0.335	0.346	0.354	50%	2.00
24	0.224	0.171	0.189	0.195	0.029	2
25	0.276	0.275	0.277	0.276	0.04	2
26	0.250	0.253	0.253	0.252	0.004	2
27	0.218	0.220	0.227	0.222	0.00496	2
28	0.400	0.310	0.290	0.333	0.117	2
29	ND	ND	ND	ND	0.02	2.06
30	0.265	0.264	0.260	0.263	0.009	2
31	0.266	0.278	0.289	0.278	0.041	2
32	0.291	0.288	0.284	0.288	0.086	2
33	0.260	0.286	0.276	0.274	0.051	2
34	0.380	0.385	0.384	0.383	0.004	2
35	0.298	0.306	0.310	0.305	0.2	2

"---" denotes data/information was not provided by participants

"ND" denotes not detected

TABLE II: Participants' Results for Cadmium (Cont'd)

Lab. Code	Concentration (mg/kg)				Measurement Uncertainty	
	Data #1	Data #2	Data #3	Mean	Expanded Uncertainty (mg/kg)	Coverage Factor
36	0.369	0.310	0.456	0.378	0.110	0.220
37	0.364	0.367	0.362	0.364	0.011	2
38	ND	ND	ND	ND	---	---
39	0.283	0.291	0.280	0.285	0.014	2
40	0.389	0.387	0.389	0.388	0.097	2
41	0.250	0.239	0.240	0.243	0.0035	2.160
42	0.151	0.162	0.170	0.161	0.028	2
43	0.210	0.228	0.240	0.226	0.0452	2
44	0.680	0.667	0.678	0.675	0.047	2
45	0.212	0.208	0.201	0.207	0.0037	2
46	0.273	0.226	0.282	0.260	0.017	2
47	0.285	0.278	0.285	0.283	0.008	2
48	0.182	0.185	0.180	0.182	0.0118	2
49	0.375	0.377	0.368	0.373	0.0095	2
50	0.283	0.265	0.261	0.270	0.02	2.0
51	0.267	0.263	---	0.265	0.053	2
52	0.286	0.292	0.276	0.285	0.060	2
53	0.430	0.410	0.420	0.420	---	---
54	0.291	0.294	0.269	0.285	0.021	2
55	0.162	0.185	0.167	0.171	0.019	2.78
56	0.278	0.298	0.288	0.288	0.009	2
57	0.310	0.304	0.310	0.308	0.003	2
58	0.269	0.277	0.285	0.277	0.008	2
59	0.273	0.261	0.242	0.259	0.059	2
60	<0.4	<0.4	<0.4	<0.4	0.112	2
61	0.271	0.267	0.267	0.268	0.019	2
62	0.198	0.124	0.161	0.161	0.075	2
63	1.60	1.60	1.70	1.60	0.40	2
64	<0.50	<0.50	<0.50	<0.50	---	---
65	0.278	0.271	0.275	0.275	0.058	2
66	0.291	0.312	0.288	0.297	0.026	2
67	0.258	0.247	0.261	0.255	0.00624	2
68	0.242	0.261	0.289	0.264	0.0050	2
69	0.251	0.252	0.259	0.254	0.021	2
70	0.29	0.30	0.30	0.30	0.09	2

"---" denotes data/information was not provided by participants

"ND" denotes not detected

TABLE II: Participants' Results for Cadmium (Cont'd)

Lab. Code	Concentration (mg/kg)				Measurement Uncertainty	
	Data #1	Data #2	Data #3	Mean	Expanded Uncertainty (mg/kg)	Coverage Factor
71	0.297	0.314	0.309	0.307	0.11	2
72	0.293	0.282	0.285	0.287	0.028	2
73	0.27	0.26	0.25	0.26	0.04	2
74	0.365	0.375	0.358	0.366	0.0022389	2
75	0.294	0.279	0.290	0.29	0.02	1
76	0.269	0.279	0.279	0.276	0.069	2
77	0.260	0.265	0.255	0.260	0.05	2
78	0.235	0.245	0.241	0.240	0.004	2
79	0.263	0.266	0.267	0.265	0.027	2
80	0.158	0.174	0.179	0.170	0.120 c	2
81	0.30	---	---	0.30	0.03	2
82	0.22	0.23	0.23	0.23	0.035	2
83	0.284	0.285	0.285	0.285	0.044	2
84	0.272	0.266	0.262	0.267	0.043	2
85	0.283	0.279	0.289	0.283	0.016	2
86	0.278	0.272	0.272	0.274	---	---
87	0.287	0.268	0.271	0.275	0.044	2
88	0.28	0.23	0.22	0.24	0.04	2
89	0.208	0.207	0.216	0.210	0.05	2
90	2.48	2.16	2.28	2.31	0.36	2
91	<0.540	<0.540	<0.540	<0.540	0.460	2
93	0.348	0.361	0.363	0.357	0.016	2
94	0.307	0.311	0.305	0.308	0.0271	2
95	0.225	0.209	0.210	0.215	0.003	2
96	0.287	0.280	0.275	0.281	---	---
97	0.305	0.292	0.296	0.298	0.014	2
98	0.271	0.229	0.274	0.258	0.07	2.0
99	0.63	0.61	0.61	0.62	0.12	2
100	0.247	0.252	0.272	0.257	0.0077	2
101	0.268	0.27	0.27	0.27	0.42	---
102	0.1050	0.1150	0.1000	0.1067	---	---

"---" denotes data/information was not provided by participants

"ND" denotes not detected

"c" denotes mean concentration

TABLE III: Participants' Results for Lead

Lab. Code	Concentration (mg/kg)				Measurement Uncertainty	
	Data #1	Data #2	Data #3	Mean	Expanded Uncertainty (mg/kg)	Coverage Factor
1	1.16	1.04	1.15	1.1	0.5	2
2	0.476	0.453	0.476	0.468	0.1	95%
3	1.35	1.14	1.17	1.22	0.32	2
4	1.51	1.59	1.54	1.55	0.12	2.776
5	ND	ND	ND	ND	---	---
6	1.72	1.56	1.54	1.61	0.208	2
7	1.47	1.59	1.51	1.52	0.31	2
8	1.710	1.620	1.740	1.690	0.118	2
9	1.50	1.48	1.51	1.50	0.034	6
10	1.57	1.51	1.43	1.50	0.140	2
11	63.3	67.3	61.6	64.1	1.7	1.65
12	1.33	1.31	1.33	1.32	0.1980	2
13	1.750	1.704	1.188	1.547	0.317	2
14	1.26	1.39	1.47	1.37	0.29	2
15	1.48	1.50	1.49	1.49	0.21	2
16	1.120	1.122	1.115	1.119	0.223	2
17	1.451	1.413	1.431	1.432	0.236	2
18	1.58	1.30	1.11	1.33	0.50	2
19	1.549	1.554	1.518	1.540	0.039	2
20	1.18	1.16	1.19	1.18	0.12	2
21	45.491	41.355	45.160	44.0	5	2
22	1.391	1.423	---	1.407	0.017	2
23	2.49	2.43	2.33	2.42	4.5%	2.00
24	1.222	1.298	1.317	1.279	0.130	2
25	1.42	1.46	1.42	1.43	0.21	2
26	1.344	1.356	1.355	1.352	0.014	2
27	0.502	0.581	0.538	0.540	0.0396	2
28	0.090	0.100	0.096	0.095	0.010	2
29	ND	ND	ND	---	0.02	2.06
30	1.242	1.224	1.236	1.234	0.089	2
31	1.61	1.41	1.46	1.49	0.20	2
32	1.38	1.40	1.45	1.41	0.42	2
33	1.67	1.74	1.71	1.71	0.20	2
34	1.714	1.670	1.660	1.681	0.004	2
35	5.267	4.678	5.178	5.041	3.0	2

"---" denotes data/information was not provided by participants

"ND" denotes not detected

TABLE III: Participants' Results for Lead (Cont'd)

Lab. Code	Concentration (mg/kg)				Measurement Uncertainty	
	Data #1	Data #2	Data #3	Mean	Expanded Uncertainty (mg/kg)	Coverage Factor
36	1.622	1.468	1.310	1.467	0.350	0.700
37	1.691	1.648	1.582	1.640	0.076	2
38	2.672	3.054	2.926	2.884	0.179	2
39	1.292	1.332	1.296	1.307	0.055	2
40	1.652	1.687	1.563	1.634	0.487	2
41	1.43	1.43	1.42	1.43	0.244	2.160
42	1.30	1.22	1.36	1.29	0.12	2
43	0.462	0.480	0.501	0.481	0.0962	2
44	7.30	7.26	7.33	7.30	0.511	2
45	0.516	0.511	0.504	0.510	0.0036	2
46	1.23	1.19	1.29	1.24	0.028	2
47	1.61	1.72	1.77	1.70	0.164	2
48	1.75	1.76	1.71	1.74	0.116	2
49	3.60	3.50	3.57	3.56	0.1026	2
50	2.6	2.3	2.3	2.4	0.35	2
51	1.39	1.41	---	1.40	0.28	2
52	1.336	1.352	1.360	1.349	0.074	2
53	1.186	0.985	1.124	1.098	---	---
54	1.027	1.002	1.010	1.013	0.021	2
55	1.538	1.434	1.309	1.427	0.317	2
56	1.574	1.542	1.568	1.561	0.059	2
57	1.637	1.695	1.733	1.688	0.010	2
58	1.48	1.50	1.44	1.47	0.03	2
59	1.431	1.455	1.330	1.405	0.280	2
60	1.566	1.593	1.570	1.576	0.426	2
61	1.408	1.404	1.423	1.412	0.140	2
62	8.265	7.037	6.730	7.344	0.940	2
63	ND	ND	ND	ND	---	---
64	<1.00	<1.00	<1.00	<1.00	---	---
65	1.46	1.47	1.40	1.44	0.32	2
66	1.600	1.573	1.502	1.558	0.101	2
67	1.443	1.521	1.665	1.543	0.353	2
68	1.237	1.293	1.215	1.248	0.040	2
69	1.48	1.48	1.46	1.47	0.12	2
70	1.60	1.68	1.65	1.64	0.49	2

"---" denotes data/information was not provided by participants

"ND" denotes not detected

TABLE III: Participants' Results for Lead (Cont'd)

Lab. Code	Concentration (mg/kg)				Measurement Uncertainty	
	Data #1	Data #2	Data #3	Mean	Expanded Uncertainty (mg/kg)	Coverage Factor
71	2.89	2.80	2.74	2.81	0.97	2
72	1.427	1.388	1.396	1.404	0.146	2
73	1.08	1.17	1.12	1.12	0.15	2
74	2.240	2.130	2.384	2.251	0.13103	2
75	1.402	1.387	1.415	1.40	0.04	1
76	1.57	1.60	1.56	1.576	0.473	2
77	5.72	5.78	5.70	5.73	0.40	2
78	1.46	1.56	1.39	1.47	0.07	2
79	1.507	1.522	1.538	1.522	0.107	2
80	1.200	1.240	1.249	1.230	0.120 c	2
81	1.6	---	---	1.6	0.1	2
82	1.36	1.36	1.38	1.37	0.022	2
83	1.343	1.367	1.363	1.358	0.155	2
84	1.499	1.527	1.472	1.499	0.225	2
85	1.467	1.502	1.490	1.486	0.082	2
86	1.211	1.167	1.219	1.199	---	---
87	1.56	1.52	1.60	1.56	0.312	2
88	1.33	1.08	1.06	1.16	0.10	2
89	1.37	1.36	1.31	1.35	0.2	2
90	7.21	6.47	6.85	5.85	0.36	2
91	<6.05	<6.05	<6.05	<6.05	0.780	2
93	1.02	0.949	0.918	0.962	0.105	2
94	1.65	1.57	1.61	1.61	0.142	2
95	1.066	1.161	1.114	1.114	0.004	2
96	1.652	1.630	1.618	1.633	1.2	2
97	1.081	1.109	1.101	1.097	0.154	2
98	2.60	2.36	2.04	2.33	0.05	2.0
99	1.67	1.62	1.60	1.63	0.33	2
100	1.37	1.37	1.37	1.37	0.0006	2
101	1.3	1.28	1.3	1.3	0.89	---
102	1.050	1.200	1.100	1.112	---	---

"---" denotes data/information was not provided by participants

"ND" denotes not detected

"c" denotes mean concentration

TABLE IV: Participants' Methodologies & Techniques

Lab Code	Analytical Instrument	Digestion method	Digestion medium	Digestion condition(s)	Method of Calibration
1	ICP-OES	Heating / wet ashing	HNO_3:$HClO_4$	205℃	SCC
2	ICP-MS	Microwave	HNO_3/HCl	200℃	SCC
3	ICP-AES / ICP-OES	Microwave	HNO_3/H_2O_2	120-175℃	SCC
4	ICP-MS	Heating	HNO_3	hot block digestor	SCC
5	FAAS	Microwave	HNO_3/H_2O_2	150-190℃	SCC
6	ICP-MS	Microwave	Aqua regia	805W 80.2bar	SCC
7	ICP-MS	Microwave	HNO_3/H_2O_2	160℃	SCC
8	ICP-MS	Microwave	HNO_3/H_2O_2	100-190℃	SCC
9	ICP-MS	Microwave	HNO_3/H_2O_2	120-180℃	SCC
10	ICP-MS	Wet ashing	HNO_3	95℃	SCC
11	ICP-AES	Wet ashing	HNO_3/$HClO_4$	heating until clear	SA
12	ICP-MS	Wet digestion / eating	HNO_3/HCl	85℃	SCC
13	FAAS	Dry Ashing	Furnace	100-450℃	SCC
14	GFAAS	Microwave	HNO_3	200℃	SCC
15	ICP-MS	Microwave	HNO_3	85-190℃	SCC
16	GFAAS	Heating/ wet oxidation	HNO_3	80℃	SCC
17	ICP-MS	Wet ashing	HNO_3	50-130℃	SCC
18	GFAAS	Wet digestion	HNO_3/HCl/H_2O_2	200℃	SCC
19	GFAAS	Wet ashing	HNO_3/H_2O_2	105℃	SA
20	ICP-MS	Hot wet acid digestion	HNO_3/HF	100℃	SCC
21	FAAS	Dry Ashing	---	525℃	SCC
22	GFAAS	Microwave	HNO_3	200℃	SCC
23	GFAAS	Microwave	HNO_3	250-600W	SCC
24	GFAAS	Dry Ashing	---	500℃	SCC
25	ICP-MS / ICP-AES	Wet digestion	HNO_3	100℃	SCC
26	ICP-MS	Microwave	HNO_3	160℃	SCC
27	ICP-OES	Microwave	HNO_3/H_2O_2	220℃	SCC
28	ICP-AES / GFAAS	Dry Ashing/ wet ashing	HNO_3/HCl	325℃	SCC / SPC
29	ICP-OES	Microwave	HNO_3/HF/H_2O_2	180℃	SCC/ SA/ SPC
30	GFAAS	Microwave	HNO_3	0.28-1.17MPa	SCC
31	GFAAS	Microwave	HNO_3/H_2O_2/HCl	900W	SCC
32	ICP-MS	Wet ashing / Microwave	HNO_3/H_2O_2	250-600W	SCC
33	GFAAS	Wet ashing	HCl/HNO_3	110℃	SCC
34	ICP-MS	Microwave	HNO_3	20-150 psi	SCC
35	ICP-OES	Microwave	HNO_3/H_2O_2	200℃	SCC

"---" denotes data/information was not provided by participants
"SA" denotes standard addition
"SCC" denotes standard calibration curve
"SPC" denotes single point calibration

TABLE IV: Participants' Methodologies & Techniques (Cont'd)

Lab Code	Analytical Instrument	Digestion method	Digestion medium	Digestion condition(s)	Method of Calibration
36	FAAS	Dry ashing	$HNO_3/HCl/H_2O$	150℃	SCC
37	GFAAS	Wet ashing	$HNO_3/HClO_4$	250℃	SA
38	FAAS	Microwave	HNO_3/H_2O_2	180℃	SCC
39	ICP-MS	Microwave	HNO_3/HF	200℃	Internal Std. Method
40	ICP-MS	Microwave	HNO_3	180℃	SCC
41	ICP-MS	Microwave	HNO_3	180℃	SCC
42	ICP-MS	Wet ashing	HNO_3	150℃	SA
43	ICP-OES	Dry ashing	$HNO_3/HClO_4$	475℃	SCC
44	ICP-AES	Hot plate	HNO_3/HF	120℃	SCC
45	GFAAS	Microwave	HNO_3/H_2O_2	120-190℃	SCC
46	ETAAS	Microwave	HNO_3/H_2O_2	160℃	SCC
47	FAAS	Wet ashing	HNO_3/H_2O_2	180℃	SCC
48	ICP-MS	Wet ashing	HNO_3	Hot plate	SCC
49	ICP-OES	Microwave	$HNO_3/H_2O_2/H_2O$	85-210℃	SCC
50	GFAAS	Microwave	HNO_3	220℃	SCC
51	ICP-MS	Microwave	HNO_3/H_2O_2	190℃	SPC
52	ICP-OES	Dry ashing	HCl/HNO_3	450℃	SCC
53	FAAS	Wet ashing	$HNO_3/HClO_4/H_2O$	90℃	SCC
54	GFAAS	Wet ashing	$HNO_3/HCl/H_2O_2$, $H_2SO_4/H_2O_2/CH_3OH$	95-180℃	SCC
55	ICP-MS	Microwave	HNO_3/H_2O_2	100-180℃	SCC
56	ICP-MS	Heating	HNO_3	50-220℃	SCC
57	ICP-MS	Microwave	HNO_3/H_2O_2	70℃	SCC
58	ICP-MS	Wet ashing	$HNO_3/HClO_4$	100-120℃	SCC
59	ICP-MS	Microwave	HNO_3/H_2O_2	75-180℃	SCC
60	ICP-MS	Microwave	HNO_3	75-180℃	SCC
61	GFAAS	Microwave	HNO_3	50-200℃	SCC
62	FAAS	Dry ashing	HCl	550℃	SCC
63	FAAS	Dry ashing	HCl/HNO_3	---	SCC
64	FAAS	Wet ashing	$HNO_3/HCl/H_2O_2$	Heating without boiling	SCC
65	ICP-MS	Microwave	HNO_3/HCl	---	SCC
66	ICP-MS	Microwave	HNO_3/H_2O_2	200℃	SCC
67	ICP-OES	Heating	HNO_3	91-96℃	SCC
68	ICP-AES	Dry ashing	HCl/HNO_3	550℃ ,Heat to boiling	SCC
69	GFAAS	Microwave	HNO_3	120-185℃	SCC
70	ICP-MS	Microwave	HNO_3	75-180℃	SCC

"---" denotes data/information was not provided by participants
"SA" denotes standard addition
"SCC" denotes standard calibration curve
"SPC" denotes single point calibration

TABLE IV: Participants' Methodologies & Techniques (Cont'd)

Lab Code	Analytical Instrument	Digestion method	Digestion medium	Digestion condition(s)	Method of Calibration
71	ICP-OES-Pb GFAAS-Cd	Wet ashing	$HNO_3/HCl/H_2O_2$	100℃	SCC
72	GFAAS	Microwave	HNO_3	130-180℃	SCC
73	GFAAS	Microwave	HNO_3	200℃	SPC
74	FAAS	Dry ashing	HNO_3/HCl	440℃	SCC
75	ICP-MS	Microwave	HNO_3/HF	210℃	SCC
76	GFASS	Microwave	HNO_3/HCl	200-220℃	SCC / SA
77	GFASS	Microwave	HNO_3/HCl	200℃	SCC
78	GFASS	Microwave	HNO_3/ H_2O_2	75-190℃	SCC
79	ICP-MS	Microwave	HNO_3/HCl	75-190℃	SCC
80	ICP-MS	Microwave	HNO_3	85-190℃	SCC
81	ICP-MS	Microwave	HNO_3	180℃	SA
82	ICP-MS	Microwave	HNO_3	180℃	SCC
83	GFAAS	Microwave	HNO_3	---	SCC
84	ICP-MS	Microwave	$HNO_3/HCl/H_2O_2$	900w	SCC
85	ICP-MS	Microwave	HNO_3	50W	SCC
86	GFAAS	Microwave	HNO_3	160℃	SCC
87	GFAAS	Microwave	H_2O_2/HNO_3	600W	SCC
88	GFAAS	Microwave / Wet ashing	HNO_3/H_2O_2	250-600W	SCC
89	GFAAS	Microwave	HNO_3	30-80 psi	SCC
90	FAAS	Wet ashing	HNO_3	95℃	SCC
91	FAAS	Acid heating plate & calcination	Acid	---	Calibration chart with standard certificate
93	GFAAS	Wet ashing	$H_2O_2/HNO_3/HCl$	125℃	SCC
94	ICP-MS	Microwave	HNO_3/H_2O_2	210℃	SCC
95	GFAAS	Dry ashing	HNO_3	600℃	SPC
96	ICP-MS	Microwave	HNO_3	150℃	SCC
97	ICP-MS	Dry ashing/ Microwave	HNO_3	450℃(DA), 180℃(M)	SCC
98	ICP-OES	Wet ashing	HNO_3	Hot plate	SCC
99	GFAAS	Microwave	HNO_3	210℃	SA
100	ICP-MS	Wet ashing	HNO_3/H_2SO_4	320℃	SCC
101	ICP-AES	Wet ashing	HNO_3	150℃	SCC
102	FAAS	Dry ashing	HNO_3	100-600℃	SCC

"---" denotes data/information was not provided by participants
"SA" denotes standard addition
"SCC" denotes standard calibration curve
"SPC" denotes single point calibration

TABLE V: Method Recovery & Accreditation Status

Lab. Code	Recovery (%)	Correction for Recovery	Method Accreditation
1	95,91	No	No
2	---	No	Yes
3	---	No	Yes
4	Cd: 91, Pb: 140	No	Yes
5	Cd: 86.17, Pb: 87.83	No	Yes
6	---	No	No
7	---	No	Yes
8	---	No	Yes
9	---	No	Yes
10	Cd: 92, Pb:100; NIST 1573, Cd: 87%	No	Yes
11	---	No	No
12	Cd: 112, Pb: 96	No	No
13	92	No	Yes
14	130	No	No
15	100.7	No	Yes
16	---	No	No
17	---	No	Yes
18	120	Yes	Yes
19	Cd: 106, Pb: 117; NIST SRM 8436, Cd:89, Pb: 98.	No	No
20	75-125	No	Yes
21	85-115	No	No
22	Cd: 83, Pb: 65	Yes	No
23	---	No	No
24	---	No	No
25	Pb: 92-104, Cd: 90-98	Yes	Yes
26	Cd: 86.67, Pb: 94.52	No	Yes
27	90-105	No	No
28	99	Yes	Yes
29	Pb: 91.48, Cd: 97.02	---	---
30	Pb: 97.0, Cd: 94.2	No	Yes
31	Pb: 91, Cd: 94	Yes	Yes
32	~100	No	Yes
33	Cd: 97, Pb: 103	No	No
34	Cd: 102.5, Pb: 110.8	No	No
35	80	No	No

"---" denotes data/information was not provided by participants

TABLE V: Method Recovery & Accreditation Status (Cont'd)

Lab. Code	Recovery (%)	Correction for Recovery	Method Accreditation
36	Pb: 99.75	No	Pb: Yes, Cd: No
37	Pb: 96.9, Cd: 110.2	No	Yes
38	98.747, 98.144	Yes	Yes
39	Cd: 105, Pb: 97	No	No
40	Cd: 106, Pb: 91	No	Yes
41	Cd: 86.8, 87; Pb: 102.3, 104.1	No	Yes
42	Pb: 51, Cd: 43	Yes	No
43	92	No	No
44	90	No	No
45	Pb: 95.7, Cd: 94.9	No	No
46	---	No	Yes
47	Cd: 98.1 , Pb: 112	No	Yes
48	Cd: 89, Pb:104	No	No
49	---	No	Yes
50	---	No	Yes
51	---	No	Yes
52	Cd: 86.8, Pb: 95.6	No	Yes
53	---	No	No
54	---	No	No
55	---	No	Yes
56	95	No	Yes
57	101.6	Yes	Yes
58	Cd: 96.8, Pb: 92.1	No	No
59	80-120	No	Yes
60	Cd: 94.2, Pb: 104.0	No	Yes
61	Pb: 95.0, Cd: 107.0	No	Yes
62	---	No	Yes
63	---	No	Yes
64	82-108	No	Yes
65	---	No	Yes
66	SRM: Cd: 108.6, Pb: 105.1; Spike: Cd: 90.6, Pb: 95.2	No	No
67	---	No	No
68	---	No	No
69	99.3	No	Yes
70	85-110	No	Yes

"---" denotes data/information was not provided by participants

TABLE V: Method Recovery & Accreditation Status (Cont'd)

Lab. Code	Recovery (%)	Correction for Recovery	Method Accreditation
71	96	No	No
72	Cd: 102.08, Pb: 102.62	No	Yes
73	100	No	Yes
74	---	No	No
75	---	No	No
76	90	No	Yes
77	90 – 95	Yes	No
78	Cd: 98, Pb: 94	No	Yes
79	85	No	Yes
80	90-110	No	No
81	---	No	No
82	Cd: 15.4, Pb: 16.3	No	No
83	Cd: 107, Pb: 97	No	Yes
84	---	No	Yes
85	90	No	Yes
86	Cd: 102.44, Pb: 95.02	No	No
87	Cd: 98, Pb: 93	No	Yes
88	---	No	No
89	,Cd: 77, Pb: 88	No	No
90	Cd: 100, Pb: 116	Yes	No
91	Cd: 94.62, Pb: 104.29	---	---
93	Cd: 110.4, Pb: 93.2	Yes	Yes
94	102	No	No
95	>90	No	No
96	---	No	Yes (Pb only)
97	---	No	No
98	Cd: 90.6, Pb: 86.0	No	No
99	---	No	Yes
100	101.9	No	Yes
101	Cd: 98.6, Pb: 95.3	No	No
102	Cd: 85.01, Pb: 80.24	No	No

"---" denotes data/information was not provided by participants

TABLE VI: z-Scores of Participants

Lab Code	Cadmium (mg/kg)	z-score	Lead (mg/kg)	z-score
1	0.31	0.56	1.1	-1.8
2	0.275	-0.09	0.468	**-4.6**
3	0.271	-0.16	1.22	-1.3
4	0.261	-0.35	1.55	0.19
5	0.17	-2.0	<1	NA
6	0.255	-0.46	1.61	0.46
7	0.280	0	1.52	0.06
8	0.283	0.06	1.690	0.82
9	0.249	-0.57	1.50	-0.03
10	0.267	-0.24	1.50	-0.03
11	11.1	**201**	64.1	**278**
12	0.294	0.27	1.32	-0.83
13	0.330	0.94	1.547	0.18
14	0.41	2.4	1.37	-0.61
15	0.278	-0.03	1.49	-0.07
16	0.225	-1.0	1.119	-1.7
17	0.277	-0.05	1.432	-0.33
18	0.207	-1.4	1.33	-0.78
19	0.259	-0.39	1.540	0.15
20	0.246	-0.63	1.18	-1.5
21	3.22	**55**	44.0	**189**
22	0.297#	0.32	1.407#	-0.44
23	0.354	1.4	2.42	**4.1**
24	0.195	-1.6	1.279	-1.0
25	0.276	-0.07	1.43	-0.34
26	0.252	-0.52	1.352	-0.69
27	0.222	-1.1	0.540	**-4.3**
28	0.333	0.99	0.095	**-6.3**
29	<0.5	NA	<4.5	NA
30	0.263	-0.31	1.234	-1.2
31	0.278	-0.03	1.49	-0.07
32	0.288	0.15	1.41	-0.43
33	0.274	-0.11	1.71	0.90
34	0.383	1.9	1.681	0.78
35	0.305	0.47	5.041	**16**

i. Unless otherwise specified, the reported concentration (mg/kg) represents the mean of three replicate analyses
ii. "#" denotes the mean value of duplicate analyses
iii. "^" denotes only one data was received
iv. "NA" denotes not applicable
v. Unsatisfactory z-scores (ie. > ± 3) are bolded

TABLE VI: z-Scores of Participants (Cont'd)

Lab Code	Cadmium (mg/kg)	z-score	Lead (mg/kg)	z-score
36	0.378	1.8	1.467	-0.18
37	0.364	1.6	1.640	0.59
38	<0.011	NA	2.884	**6.1**
39	0.285	0.10	1.307	-0.89
40	0.388	2.0	1.634	0.57
41	0.243	-0.68	1.43	-0.34
42	0.161	-2.2	1.29	-0.96
43	0.226	-1.0	0.481	**-4.6**
44	0.675	**7.4**	7.30	**26**
45	0.207	-1.4	0.510	**-4.4**
46	0.260	-0.37	1.24	-1.2
47	0.283	0.06	1.70	0.86
48	0.182	-1.8	1.74	1.0
49	0.373	1.7	3.56	**9.1**
50	0.270	-0.18	2.4	**4.0**
51	0.265#	-0.27	1.40#	-0.47
52	0.285	0.10	1.349	-0.70
53	0.420	2.6	1.098	-1.8
54	0.285	0.10	1.013	-2.2
55	0.171	-2.0	1.427	-0.35
56	0.288	0.15	1.561	0.24
57	0.308	0.53	1.688	0.81
58	0.277	-0.05	1.47	-0.16
59	0.259	-0.38	1.405	-0.45
60	<0.4	NA	1.576	0.31
61	0.268	-0.22	1.412	-0.42
62	0.161	-2.2	7.344	**26**
63	1.60	**25**	<0.001	NA
64	<0.50	NA	<1.00	NA
65	0.275	-0.09	1.44	-0.30
66	0.297	0.32	1.558	0.23
67	0.255	-0.46	1.543	0.16
68	0.264	-0.29	1.248	-1.1
69	0.254	-0.48	1.47	-0.16
70	0.30	0.38	1.64	0.59

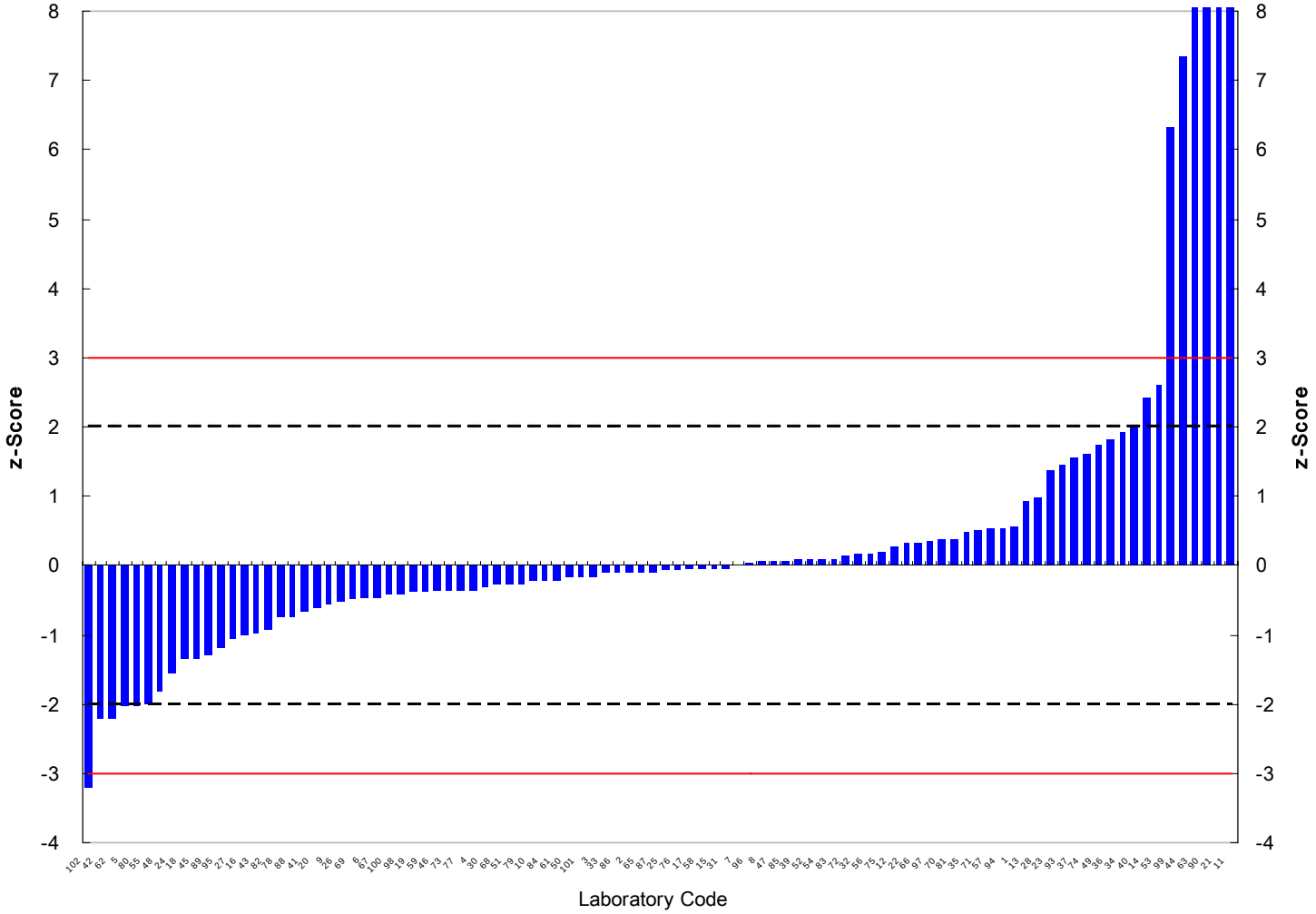
i. Unless otherwise specified, the reported concentration (mg/kg) represents the mean of three replicate analyses
ii. "#" denotes the mean value of duplicate analyses
iii. "^" denotes only one data was received
iv. "NA" denotes not applicable
v. Unsatisfactory z-scores (ie. > ± 3) are bolded

TABLE VI: z-Scores of Participants (Cont'd)

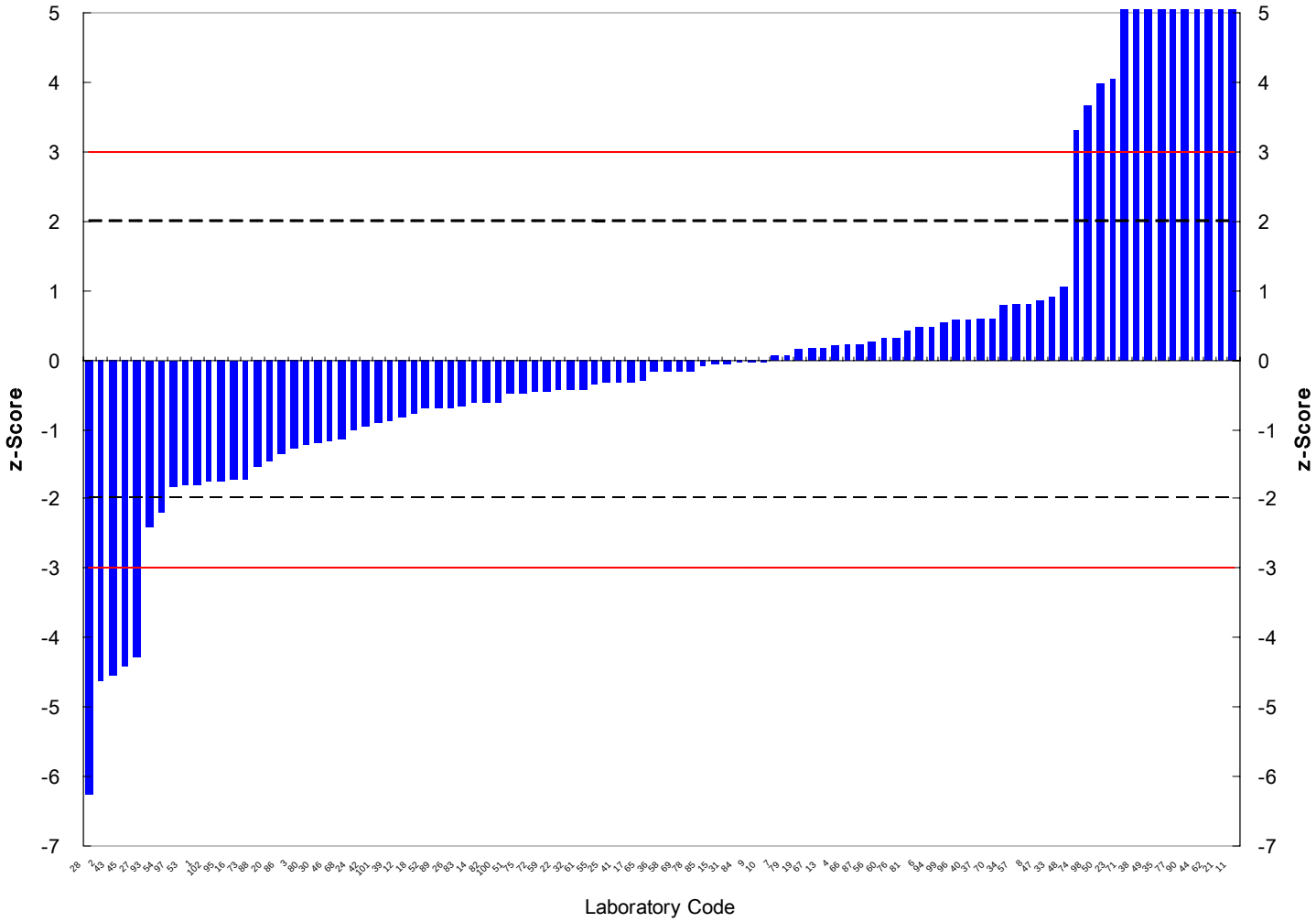
Lab Code	Cadmium (mg/kg)	z-score	Lead (mg/kg)	z-score
71	0.307	0.51	2.81	**5.8**
72	0.287	0.14	1.404	-0.46
73	0.26	-0.37	1.12	-1.7
74	0.366	1.6	2.251	**3.3**
75	0.29	0.19	1.40	-0.47
76	0.276	-0.07	1.576	0.31
77	0.260	-0.37	5.73	**19**
78	0.240	-0.74	1.47	-0.16
79	0.265	-0.27	1.522	0.07
80	0.170	-2.0	1.230	-1.2
81	0.30^	0.38	1.6^	0.42
82	0.23	-0.92	1.37	-0.61
83	0.285	0.10	1.358	-0.66
84	0.267	-0.24	1.499	-0.03
85	0.283	0.06	1.486	-0.09
86	0.274	-0.11	1.199	-1.4
87	0.275	-0.09	1.56	0.24
88	0.24	-0.74	1.16	-1.5
89	0.210	-1.3	1.35	-0.70
90	2.31	**38**	5.85	**19**
91	<0.540	NA	<6.05	NA
93	0.357	1.4	0.962	-2.4
94	0.308	0.53	1.61	0.46
95	0.215	-1.2	1.114	-1.7
96	0.281	0.02	1.633	0.56
97	0.298	0.34	1.097	-1.8
98	0.258	-0.40	2.33	**3.7**
99	0.62	**6.3**	1.63	0.55
100	0.257	-0.42	1.37	-0.61
101	0.27	-0.18	1.3	-0.92
102	0.1067	**-3.2**	1.112	-1.8

i. Unless otherwise specified, the reported concentration (mg/kg) represents the mean of three replicate analyses
ii. "#" denotes the mean value of duplicate analyses
iii. "^" denotes only one data was received
iv. "NA" denotes not applicable
v. Unsatisfactory z-scores (ie. > ± 3) are bolded

HISTOGRAM I: z-Scores of participating laboratories for Cadmium



HISTOGRAM II: z-Scores of participating laboratories for Lead



APPENDIX I: Homogeneity Test

a. A total of 17 bottles of powder were randomly selected from the bulk. Two test portions (sub-samples) of 0.5 g each were taken from each bottle for duplicate analysis of cadmium and lead in November 2006 using ICP-MS method.

b. Results of the 34 sub-samples were as follows:

Sample Code	Concentration (mg/kg) of duplicate analysis			
	Cadmium		Lead	
	#1	#2	#1	#2
15	0.263	0.263	1.465	1.448
33	0.258	0.267	1.467	1.499
39	0.270	0.262	1.471	1.451
45	0.266	0.268	1.461	1.467
68	0.258	0.266	1.415	1.465
76	0.263	0.260	1.455	1.477
100	0.262	0.260	1.495	1.519
107	0.268	0.261	1.523	1.478
112	0.260	0.264	1.471	1.468
118	0.268	0.263	1.472	1.465
123	0.261	0.266	1.433	1.460
133	0.266	0.266	1.457	1.464
161	0.265	0.265	1.449	1.461
173	0.268	0.269	1.456	1.475
204	0.261	0.261	1.448	1.412
215	0.251	0.263	1.479	1.426
236	0.257	0.258	1.422	1.458
Mean($\bar{x}$)	0.263		1.463	
SD (S_x)	0.004		0.025	
RSD(%)	1.53		1.68	

c. According to ISO13528:2005, the samples may be considered to be adequately homogeneous if:

$S_s \leq 0.3\sigma_R$

where S_s = Between samples standard deviation
σ_R = Standard deviation for proficiency assessment

σ_R (in percent) is estimated using Horwitz equation of 2 $c^{-0.15}$ where c was the mean concentration of analyte from homogeneity test:

Analyte	$\bar{x}$ (mg/kg)	σ_R (%)	$0.3\sigma_R$ (%)	$0.3\sigma_R$ (mg/kg)
Cadmium	0.263	19.41	5.82	0.0153
Lead	1.463	15.01	4.50	0.0658

APPENDIX I: Homogeneity Test (Cont'd)

S_s is calculated as:

$$\sqrt{S_x^{\,2} - (S_w^{\,2}/2)}$$

Where S_x = Standard deviation of sample averages
S_w = Within-samples standard deviation

S_w is calculated as:

$$\sqrt{\sum w_t^{\,2}/(2n)}$$

where w_t = Absolute difference of the duplicate sample
n = Number tested samples

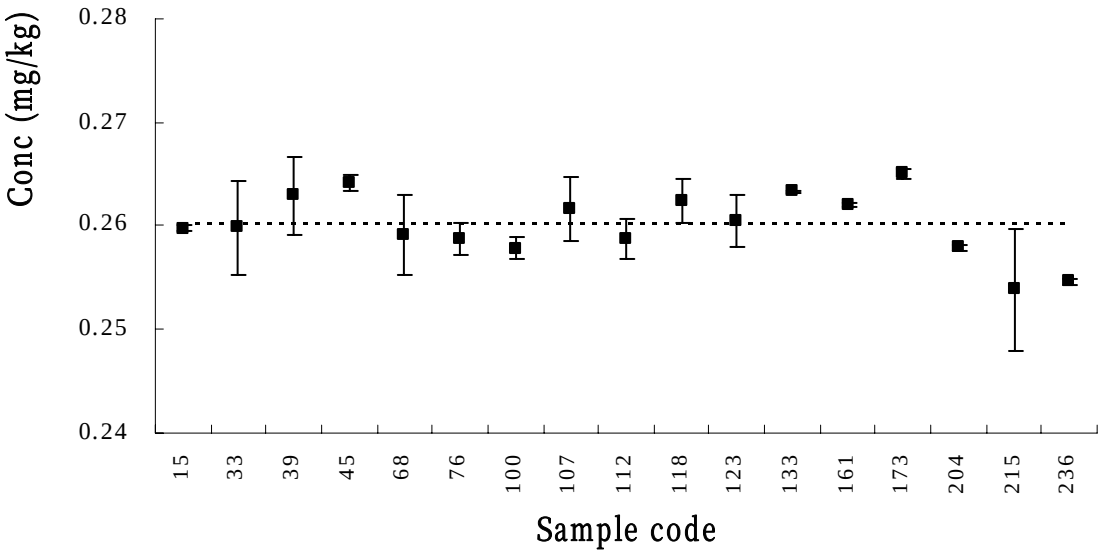
The results were summarized as follows:

Analyte	n	Σw_t^2 (mg/kg)	S_w (mg/kg)	S_x (mg/kg)	S_s (mg/kg)
Cadmium	17	4.53×10^{-4}	3.65×10^{-3}	4.03×10^{-3}	3.09×10^{-3}
Lead	17	0.0141	0.0204	0.0246	0.0199

Since the between samples standard deviations (S_s) from the homogeneity test for cadmium and lead were found to be less than that of the $0.3\sigma_R$, the materials were considered homogeneous.

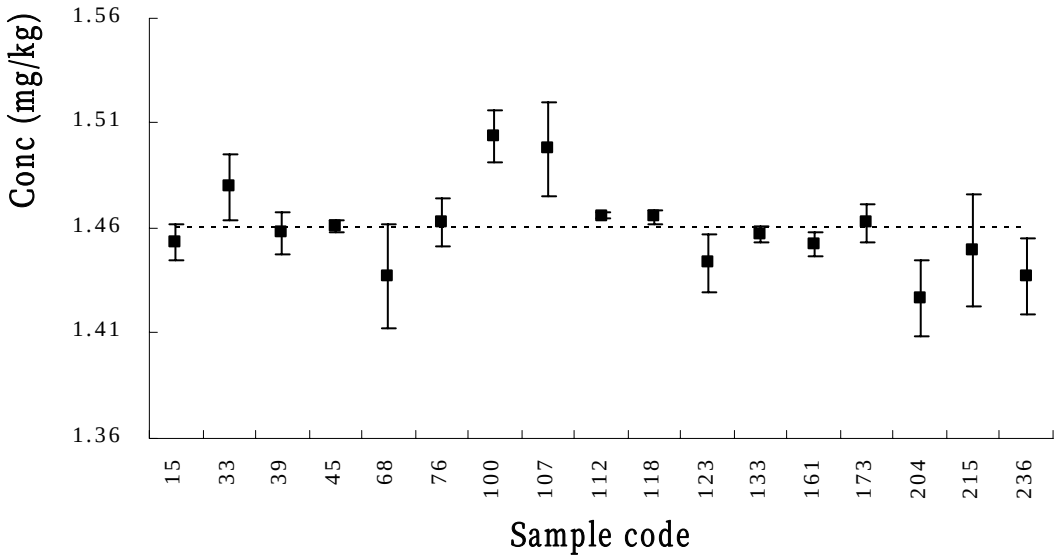
e. Graphical illustrations of random samples (n =17) in the homogeneity test

(i) Cadmium



APPENDIX I: Homogeneity Test (Cont'd)

(ii) Lead



APPENDIX II: Stability Test

a. Stability test was monitored at different intervals from December 2006 to September 2008, which has covered the time when the samples were analyzed by participants.

b. Samples selected for the stability test were stored at (i) a room temperature of 25 °C and (ii) an elevated temperature of 37 °C.

c. One sample from each temperature set was randomly selected and analyzed in duplicate or triplicate under the same operational conditions as those in homogeneity test.

d. Stability of samples was evaluated from the absolute difference between the mean value obtained in homogeneity test ($\bar{x}$) and the mean value ($\overline{yi}$) of each stability test. According to ISO13528:2005, the absolute difference should not be more than 30% of the standard deviation of the proficiency test (σ_R).

ie. $|\bar{x} - \overline{yi}| \leq 0.3\sigma_R$

e. The stability test at 25°C and 37°C was summarized below:

(i) Cadmium

Period / Temp.		Sample Code	Concentration (mg/kg)					$\lvert\bar{x} - \overline{yi}\rvert$	Status
			#1	#2	#3	$\overline{yi}$	$\bar{x}$		
Dec 06:	25°C	120	0.265	0.269	---	0.267	0.263	0.004	Pass
	37°C	132	0.272	0.267	---	0.269	0.263	0.006	Pass
Jan 07:	25°C	19	0.263	0.264	0.272	0.266	0.263	0.003	Pass
	37°C	30	0.269	0.262	0.261	0.264	0.263	0.001	Pass
Nov 07:	25°C	142	0.273	0.261	0.265	0.266	0.263	0.002	Pass
	37°C	39	0.2677	0.2697	0.2660	0.268	0.263	0.005	Pass
Sept 08:	25°C	220	0.279	0.273	0.269	0.274	0.263	0.011	Pass

APPENDIX II: Stability Test (Cont'd)

(ii) Lead

Period / Temp.		Sample Code	Concentration (mg/kg)					$\lvert\bar{x} - \bar{yi}\rvert$	Status
			#1	#2	#3	$\bar{yi}$	$\bar{x}$		
Dec 06:	25°C	120	1.417	1.423	---	1.420	1.463	0.043	Pass
	37°C	132	1.419	1.436	---	1.428	1.463	0.035	Pass
Jan 07:	25°C	19	1.489	1.458	1.517	1.488	1.463	0.025	Pass
	37°C	30	1.444	1.428	1.437	1.436	1.463	0.027	Pass
Nov 07:	25°C	142	1.445	1.420	1.453	1.439	1.463	0.024	Pass
	37°C	39	1.410	1.418	1.449	1.426	1.463	0.037	Pass
Sept 08:	25°C	220	1.483	1.452	1.463	1.466	1.463	0.003	Pass

f. Since the absolute differences between the mean value from homogeneity test ($\bar{x}$) and the mean value of the stability test ($\bar{yi}$) were found to be less than $0.3\sigma_R$ for cadmium and lead, the materials were considered to be stable at 25°C for two years and at 37°C for one year.

APPENDIX III: Instructions to Accreditation Bodies

INSTRUCTIONS
(FOR ACCREDITATION BODIES)

1. ORGANIZATION

The PT programme on "Heavy metals in food (APLAC T065)" is organized by the Hong Kong Government Laboratory (HKGL) in collaboration with Hong Kong Accreditation Service (HKAS) under the auspices of the Asia-Pacific Laboratory Accreditation Cooperation (APLAC). The main objective of the programme is to evaluate the performance of participating laboratories on the concerned tests through inter-laboratory comparison.

2. SAMPLE

Accreditation bodies (AB) should receive the PT samples and instruction information (hard copy and soft copy) from the organizers. The samples that each contains about 5 g of dried herbal powder (≤ 250 m) are properly packaged in amber bottles. Upon receipt, AB should carefully check the samples (number of bottles, physical conditions, etc.) and shall promptly acknowledge the HKGL by returning the "Receipt Form (for Accreditation Bodies)" electronically to T65@govtlab.gov.hk. New sample(s) will be replaced for any missing and damaged bottles.

AB are recommended to promptly distribute the samples, together with the "Instructions for Participating Laboratories" and "Results Proforma" (hard and soft copy), to their respective participating laboratories. The samples shall be stored in a secure environment at room temperature (around 25°C) before dispatch.

AB shall remind their respective participating laboratories carefully check the sample upon receipt and promptly acknowledge the HKGL by returning the "Receipt Form (for Participating Laboratories)" electronically to T65@govtlab.gov.hk.

3. RESULTS

AB shall inform their respective participating laboratories complete Part I and II of the "Result Proforma" and submit electronically to the HKGL on or before **31 August 2008** to T65@govtlab.gov.hk. (Notes: 1. Unless with special reasons, results submitted after the deadline might not be accepted by the organizers. 2. To avoid poor transmission quality, results by fax are not accepted.)

4. ENQUIRIES

Please contact the HKGL for further enquiries at T65@govtlab.gov.hk

APPENDIX IV: Receipt Form for Accreditation Bodies

RECEIPT FORM
(FOR ACCREDITATION BODIES)

From :
.....
(Name of AB)

.....
(Contact Person)

Email :
.....

Date:
.....

To: Dr. Yiu-chung WONG
The PT Advisory Board
HKGL,
Hong Kong.

Email: T65@govtlab.gov.hk

I hereby acknowledge the receipt of _____ # sample(s) for the APLAC T065 programme.

The sample(s) is/are found *(intact / damaged) and should be *(suitable / not suitable) for laboratory testing.

Remark(s) :
.....
.....
.....
.....
_____

Number of samples
* Please circle as appropriate

APPENDIX V: Instruction to Participating Laboratories

INSTRUCTIONS
(FOR PARTICIPATING LABORATORIES)

1. ORGANIZATION

The PT programme on "Heavy metals in food (APLAC T065)" is organized by the Hong Kong Government Laboratory (HKGL) in collaboration with Hong Kong Accreditation Service (HKAS) under the auspices of the Asia-Pacific Laboratory Accreditation Cooperation (APLAC). The main objective of the programme is to evaluate the performance of participating laboratories on the concerned tests through inter-laboratory comparison.

2. SAMPLE

(a) Participating laboratories will independently receive an amber sample bottle that containing about 5 g of herbal sample powder from the organizers or via their respective accreditation bodies (AB).

(b) Upon receipt of the sample, participating laboratories should carefully inspect the sample for any physical damages and defects.

(c) Participating laboratories shall promptly acknowledge receipt of the sample by returning the "Receipt Form (for Participating Laboratories)" electronically to the HKGL to T65@govtlab.gov.hk. New samples will be replaced for any damaged claims.

(d) Intact sample is recommended to be stored in a secure environment at room temperature (around 25°C) before use. Sample should be immediately capped after used and be kept in a dry box when not in use.

3. ANALYSIS

(a) Participating laboratories are required to determine the concentration (in **mg/kg**) of cadmium and lead in the sample. Determination should be conducted in triplicate.

(b) Participating laboratories should use their preferable test methods (could be accredited, in-house, modified, ad hoc methods, etc.) which are normally used to test their customer samples and record the method used on the results sheet.

(c) A quantity of about 0.5g of sample is recommended to use for each analysis.

APPENDIX V: Instruction to Participating Laboratories (Cont'd)

4. RESULTS

(a) Report all the triplicate data and the arithmetic mean in Part I of the "Results Proforma".

(b) Estimate and report the expanded uncertainty (± mg/kg) for individual analyte.

(c) Fill in the information of your performed analytical method in Part II of the "Results Proforma".

(d) Submit the results electronically to the HKGL on or before **31 August 2008** to T65@govtlab.gov.hk using the soft copy of the "Result Proforma" provided. (Notes: 1. Unless with special reasons, results submitted after the deadline might not be accepted by the organizers. 2. To avoid poor transmission quality, results by fax are not accepted.)

5. ENQURIES

Please contact the representative of the HKGL for further enquiries concerning the programme:

Dr. Yiu-chung WONG
PT Advisory Board, HKGL.
Email: T65@govtlab.gov.hk

APPENDIX VI: Receipt Form for Participating Laboratories

RECEIPT FORM
(FOR PARTICIPATING LABORATORIES)

From :

.....
(Name of Participating Laboratory)

.....
(Contact Person)

Email :

.....

Date:

.....

To: Dr. Yiu-chung WONG
The PT Advisory Board
Hong Kong Government Laboratory,
Hong Kong.

Email: T65@govtlab.gov.hk

I hereby acknowledge the receipt of ONE sample for the APLAC T065 programme.

The sample(s) is/are found (damaged / satisfactory)* and should be (suitable / not suitable)* for laboratory testing.

Remark(s) : .....

.....

.....

.....

.....

* Please circle as appropriate

APPENDIX VII: Results Proforma

RESULTS PROFORMA

<Both Part I & II **MUST** be completed by Participating Laboratory>

Name of Participating Laboratory: _____

_____

Contact Person / Post: _____

Part I: Analytical Results

Analyte	§Concentration (in **mg/kg**, based on as-received weight)				Measurement Uncertainty	
	Data 1	Data 2	Data 3	MEAN	Expanded Uncertainty (± mg/kg)	Coverage Factor (*k*)
Cadmium (Cd)						
Lead (Pb)						

§ Report values to 3 significant figures or 3 decimal places, whichever is appropriate

◆ Please submit this "Result Proforma" electronically to the HKGL at T65@govtlab.gov.hk

◗ 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

To be continued ...

RESULT PROFORMA

< Both Part I & II MUST be completed by Participating Laboratory >

Part II: Test Parameters

1. Analytical Instrument(s): *ICP-MS / ICP-AES / ICP-OES / FAAS / GFAAS

   Others: _____ (please specify)

2. Digestion Method: *Heating / Dry Ashing/ Wet Ashing / Microwave-assisted / ASE

   Others: _____ (please specify)

3. Digestion Medium: _____

4. Digestion Condition(s): _____

5. Method of Calibration: *Standard Cal. Curve / Single-point Calibration / Standard Addition

   Others: _____ (please specify)

6. Recovery (%) _____ %

7. Correction for Recovery? *YES / NO

8. Method accredited? *YES / NO

9. Other additional information: _____

*Please circle as appropriate