



APLAC T074

**Pb and Cd in Rice Proficiency Testing
Program**

FINAL REPORT

Feb 2012

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Summary of results

1. This report summarizes the results for the APLAC T074 proficiency testing program on the determination of Lead and Cadmium contents in rice. This program covered a series of tests on rice.
2. A total of 75 laboratories from 32 different economies participated in the proficiency testing program. 61 laboratories submitted their results in time, thus were included in the statistical analysis. (APPENDIX A)
3. Assigned value and standard deviation are determined by robust analysis according to Algorithm A in ISO 13528:2005. The z-score is used as the numerical indicator to assess individual participant's performance in the program.
4. Results of participants are summarized as follows:

Table1-1 Summary of F Sample Testing Results

Performance Achieved (F)		$ Z_F \leq 2.0$		$2.0 < Z_F < 3.0$		$ Z_F \geq 3.0$		Total
Number of Participants	Pb	51	86.4%	2	3.4%	6	10.2%	59 (100%)
	Cd	51	83.6%	9	14.8%	1	1.6%	61 (100%)

Table1-2 Summary of G Sample Testing Results

Performance Achieved (G)		$ Z_G \leq 2.0$		$2.0 < Z_G < 3.0$		$ Z_G \geq 3.0$		Total
Number of Participants	Pb	53	89.8%	3	5.1%	3	5.1%	59 (100%)
	Cd	53	86.9%	6	9.8%	2	3.3%	61 (100%)

1. Introduction

- 1.1 This report summarizes the results for the APLAC T074 proficiency testing program on the determination of Lead and Cadmium contents in rice. The program was organized by the China National Accreditation Service for Conformity Assessment (CNAS) in collaboration with Shenyang Entry-Exit Inspection & Quarantine Bureau of People's Republic of China (SYCIQ), under the auspices of Asia-Pacific Laboratory Accreditation Cooperation (APLAC).
- 1.2 SYCIQ is one site of an accredited proficiency testing provider (PT0005) by CNAS against ISO/IEC 17043:2010. SYCIQ has organized many proficiency testing programs, such as APLAC T048 and APLAC T072.
- 1.3 A total of 75 laboratories participated in the proficiency testing program. 61 laboratories submitted their results.
- 1.4 Each participant was confidentially assigned with a unique laboratory code which is used throughout the program.

2. Objectives

- 2.1 The main objective of the program was to evaluate the performance of participating laboratories in the analysis for the content of Lead and Cadmium by means of inter-laboratory comparison.
- 2.2 Test results from participants are evaluated according to the statistical techniques outlined in ISO 13528:2005 "Statistical methods for use in proficiency testing by inter laboratory comparisons". The z-score is used as a numerical indicator to assess individual participant's performance in the program.

3. Proficiency Testing Items (PTIs)

3.1 Preparation:

Two different concentrations were designed for this scheme, named F and G respectively. Each kind sample was prepared by adding designed amounts of Lead and Cadmium (Pb CAS Registering Number: 7439-92-1, Cd CAS Registering Number: 7440-43-9) in the blank rice. Then, the rice samples were grinded and mixed well to make it homogeneous. Respectively, each kind sample was divided and packed into sealed glass bottles covered with dark packaging to make into testing samples. The testing samples were randomly labeled and recorded. Testing samples packed with the Instructions for Participants (see APPENDIX E) were

distributed directly to appropriate receivers as requested, accreditation bodies or participants or other bodies..

- 3.2 Laboratories were requested to perform the test according to the “INSTRUCTION” and to record their results on the “RESULTS SHEET”, both of which were distributed to participants with the samples in October, 2011. (APPENDIX E to G)
- 3.3 All ABs were requested to read the “Instructions for Accreditation Bodies” and to redirect the sample(s) and the attached documents to their nominated laboratories as soon as possible. (APPENDIX D)
- 3.4 The samples randomly selected were determined for their homogeneity and stability. The data reflected that the samples were sufficiently homogeneous and stable. Details on homogeneity and stability could be found in APPENDIX C

4. Test Required

- 4.1 Participants were required to determine the content of Lead and Cadmium in the test sample according to their preferable test methods (that could be accredited, in-house, modified, etc.), and it is recommended to use AA, ICP-MS methods or other suitable methods. Moreover, it was strongly suggested to fill in APPENDIX H which will be provided to SYCIQ for technical comments in the Final Report.

- 4.2 Participants were asked to report results according to the Instructions.

5. Statistical Evaluation

- 5.1 Participants’ test results

Test results received from participants are presented in APPENDIX A.

- 5.2 Performance evaluation

- 5.2.1 According to Algorithm A in Annex C of ISO 13528:2005, the data are sorted into ascending order, by:

$$X_1, X_2, \dots, X_i, \dots, X_p$$

Denote the robust average and robust standard deviation of these data by x^* and s^* .

Calculate the initial values for x^* and s^* as:

$$x^* = \text{median of } x_i \quad (i=1, 2, \dots, p)$$

$$s^* = 1.483 \text{ median of } |x_i - x^*| \quad (i=1, 2, \dots, p)$$

Update the values of x^* and s^* as follows. Calculate:

$$\delta = 1.5 s^*$$

For each x_i ($i=1, 2, \dots, p$), calculate:

$$x_i^* = \begin{cases} x^* - \delta, & \text{if } x_i < x^* - \delta \\ x^* + \delta, & \text{if } x_i > x^* + \delta \\ x_i, & \text{otherwise} \end{cases}$$

Calculate the new values of x^* and s^* from:

$$x_i^* = \sum x_i^* / p$$

$$s^* = 1.134 \sqrt{\sum (x_i^* - x^*)^2 / (p-1)}$$

where the summation is over i .

The robust estimates x^* and s^* are derived by an iterative calculation, i.e. by updating the values of x^* and s^* several times using the modified data, until the process converges. Convergence is assumed when there is no change from one iteration to the next in the significant figure of the robust standard deviation and the robust average specified in the Instructions for laboratories.

When the assigned value is derived as a robust average, the standard uncertainty of the assigned value X is estimated as:

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

where s^* is the robust standard deviation;

p is the number of results.

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

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

5.2.3 The z-score is normally interpreted as follows:

$|z| \leq 2.0$ Satisfactory $2.0 < |z| < 3.0$ Warning signal $|z| \geq 3.0$ Action signal

5.2.4 Robust statistical method was employed to assess the performance of participating laboratories to implement relevant tests of Lead and Cadmium contents. As a general rule, according to ISO/IEC 17043, any absolute value of individual, z-score falls in the range of 2.0 to 3.0 is regarded as a warning result, while any absolute value of z-score exceeding 3.0 is regarded as an action result. Details are shown in APPENDIX A. Participants with $|z| \geq 3.0$ should thoroughly investigate their results and report them to their AB. Participants with a z-score in the range $2.0 < |z| < 3.0$ are encouraged to review their results.

5.2.5 Graphical Displays: In addition to tables of the results and z-score, and summary statistics, the ordered z-score histograms are included in the report. On these charts each laboratory's z-score is shown in order of magnitude, and marked with its code number of the lab. From these charts each laboratory can readily compare its performance relative to the other laboratories.

6. Results

6.1 A total of 75 laboratories participate in this program, among which 61 submitted results. 59 participants tested Lead; 61 participants tested Cadmium. Please refer to APPENDIX A for participant results and statistical analysis, and table 2 and 3 for other statistics.

Table 2 Summary of laboratories with satisfactory results, warning and action signals

Result	Lab code	Sum	Percentage (%)
Labs with all results of $ z \leq 2.0$	03、05、06、07、08、 09、11、14、15、17、 18、28、31、32、33、 34、35、36、37、38、 39、42、44、46、47、 49、50、54、55、56、 57、58、59、61、62、 63、64、67、68、69、 70、71、72	43	70.5
Labs with results of $2.0 < z < 3.0$	04、13、16、20、26*、 27、40、52、53、65、 66	11	18.0
Labs with result of $ z \geq 3.0$	01、02、10、19、41、 43、45	7	11.5

Note : * Lab 26 only tested one element Cd.

Table3 Summary statistics for z-score

Lab code	Lead		Cadmium	
	Z _F	Z _G	Z _F	Z _G
T074-01	-4.17 A	-4.38 A	-1.46	-1.41
T074-02	-0.27	-3.27 A	0.34	0.90
T074-03	-0.75	-1.33	0.28	0.22
T074-04	0.13	0.76	-2.43 W	-2.57 W
T074-05	-0.59	-1.33	0.21	0.56
T074-06	0.61	0.62	-0.43	-0.39
T074-07	-0.03	-0.21	0.34	0.22

Lab code	Lead		Cadmium	
	Z _F	Z _G	Z _F	Z _G
T074-08	-0.35	-0.49	0.67	0.22
T074-09	/	/	-1.01	-0.94
T074-10	-5.04A	-2.92W	0.02	0.42
T074-11	0.13	0.34	1.44	0.83
T074-13	-0.04	1.73	2.62 W	1.70
T074-14	1.72	0.62	-0.37	-1.14
T074-15	-0.27	0.27	-0.56	-0.63
T074-16	-0.03	-0.01	2.15 W	0.83
T074-17	-0.35	0.62	0.67	0.49
T074-18	0.45	0.97	0.99	0.83
T074-19	5.44 A	1.63	-0.31	-0.47
T074-20	-1.86	-1.28	-2.60 W	-2.43 W
T074-26	/	/	-2.17 W	-2.23 W
T074-27	2.28 W	-1.88	-1.27	1.92
T074-28	0.53	0.55	0.54	0.29
T074-31	0.05	0.34	0.99	0.77
T074-32	0.68	-0.01	0.28	-0.60
T074-33	-0.11	0.27	0.28	0.08
T074-34	-0.27	0.13	-0.11	-0.26
T074-35	-0.03	0.06	0.54	0.22
T074-36	-0.43	-0.35	0.54	0.42
T074-37	0.45	0.48	0.80	0.36
T074-38	1.36	0.83	0.80	0.31
T074-39	0.13	-0.14	-0.24	-0.94
T074-40	1.24	1.38	-1.33	2.47 W
T074-41	4.98 A	5.20 A	2.15 W	1.45
T074-42	-0.69	-0.47	-0.26	-0.33
T074-43	5.54 A	1.18	-0.69	3.56 A
T074-44	1.64	-0.77	0.15	1.24
T074-45	-3.37 A	-2.16 W	-4.05 A	-3.11 A

Lab code	Lead		Cadmium	
	Z _F	Z _G	Z _F	Z _G
T074-46	-0.35	-0.08	-0.11	-0.12
T074-47	0.05	0.34	0.47	0.22
T074-49	0.41	0.33	0.70	0.29
T074-50	0.45	0.90	0.67	0.29
T074-52	-1.07	-1.46	-2.30 W	-2.16 W
T074-53	-0.25	-0.58	-2.11 W	-2.23 W
T074-54	-1.22	-0.98	0.21	-0.19
T074-55	0.92	0.76	0.02	-0.05
T074-56	-0.35	0.20	0.02	1.45
T074-57	-1.94	-1.05	-1.33	-1.48
T074-58	-0.19	-0.08	0.09	-0.12
T074-59	-0.27	0.13	0.15	0.22
T074-61	-1.46	-1.12	-0.75	-0.53
T074-62	1.56	1.24	0.09	0.08
T074-63	-0.03	0.76	0.54	0.56
T074-64	-1.22	-0.98	0.99	0.77
T074-65	-2.50 W	-2.16 W	0.21	0.02
T074-66	0.92	0.69	2.09 W	-0.19
T074-67	-0.91	-0.42	-0.11	-0.19
T074-68	-0.11	0.06	0.47	0.22
T074-69	0.13	0.34	-1.66	-1.55
T074-70	0.45	0.55	0.21	-0.12
T074-71	-0.59	-0.35	-0.37	-0.32
T074-72	1.00	0.76	0.54	0.56

Note 1: The value marked with 'A' is Action signal, which is of $|z| \geq 3.0$; the value marked with 'W' is Warning signal, which is of $2.0 < |z| < 3.0$

6.2 Table 4 shows the robust average of PT results calculated by ISO 13528:2005.

Table 4 Summary of robust statistical results

Elements	Pb		Cd	
Sample	F	G	F	G
Robust average (mg/kg)	1.194	1.411	1.377	1.118
Standard uncertainty (mg/kg)	0.021	0.024	0.025	0.024
Robust SD (mg/kg)	0.126	0.144	0.155	0.147
RSD(%)	10.53	11.25	10.20	13.15
No. lab	59		61	

7. Comments

7.1 Technical knockout

Lab 09 reported Lead as <0.20mg/kg for F sample and <0.20mg/kg for G sample, while the robust averages are 1.19mg/kg and 1.41mg/kg. Obviously, the reported data is not proper to be included in the statistical analysis.

7.2 Technical Commentary

In order to facilitate the participants to find the reasons of action results and improve their testing performance, main factors that influence the analytical results of Lead and Cadmium contents are described as following.

- Reference materials
- Sample treatment
- Reagents
- Operating Condition
- Dilution procedure of the sample and standard solution

In this program, the standard solutions used by the participants have different sources and purities, in particular, some participants used standard solution with lower purity, and it might affect the final results.

Currently, the sample treatment methods of Lead and Cadmium, commonly include dry digestion, wet digestion and microwave digestion, etc. Every digestion method has its characteristic. For example, in the process of the dry digestion, the measured element is inclined to volatilize

with the destroying of the organic compound in the high temperature environment, which may lead to inaccurate results. For another example, in the process of wet digestion, most of the final digestion solutions are acidic, and under the acidic conditions, the detector will be disturbed, which may lead to inaccurate results.

In this program, the digestion solutions used by the participants are different, including HNO_3 , $\text{HNO}_3\text{-H}_2\text{O}_2$, $\text{HNO}_3\text{-HCl}$, $\text{HNO}_3\text{-H}_3\text{PO}_4$, and the capability for digestion are different too. For example, the usage of H_2O_2 can greatly increase the digestion efficiency. The choice of the digestion solution can influence the final digestion efficiency, which may eventually lead to the test results of large deviations.

Under some testing conditions, the sample solution should be diluted to have the equivalent concentration with the standard solution, and the dilution multiple may enlarge the volume error, which may ultimately affect the test results.

7.3 Uncertainty

For the evaluation of the uncertainty, an explication of the uncertainty was made referred to 'ISO GUM methodology' in *INSTRUCTIONS TO PARTICIPATING LABORATORIES*.

It was found that the understanding or the application of measurement uncertainty varies a lot among the participants. There are even some participants not reporting their measurement uncertainty at all. On one hand, some reported uncertainties just based the uncertainty of the mean from the 4 replicates, not covering all the influencing parameters in the measurement. On the other hand, some uncertainty is larger than the robust standard deviation of this scheme and this is unreasonable according to the international practice.

8. Remarks

Efforts and supports from all ABs and participants to this program are gratefully noted. Participants are welcome to forward any comments on this PT to the following information:

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9. References

- [1] ISO/IEC 17043: 2010, "Conformity assessment -- General requirements for proficiency testing".
- [2] ISO 13528:2005, "Statistical methods for use in proficiency testing by interlaboratory comparisons".
- [3] ISO-Guide to the Expression of Uncertainty in Measurement.

APPENDIX A

Summary of Testing Results

The numbers of laboratories with respect to their accreditation bodies are listed in Table A.1.

The testing results on Lead and Cadmium contents obtained from the participants with the z-scores are listed in Table A.2~Table A.5

In these tables, some abbreviations and symbols were used. The symbols Z_F and Z_G denote individual z-scores of result F, result G, respectively. The symbol "W" indicates a z-score derived from a warning result, while "A" follows a z-score derived from an action result.

Table A.1 Summary of Participants

Economy	Accreditation Body	Number of Labs	Number of Labs Reported
Australia	National Association of Testing Authorities(NATA)	4	4
Brazil	CGCRE-Coordenaco Geral da Acreditaco;INMETRO-Instituto Nacional de Metrologia,Qualidade e Tecnologia	2	0
Canada	Standards Council of Canada	4	2
China	China National Accreditation Service for Conformity Assessment (CNAS)	6	6
Chile	INSTITUTO NACIONAL DE NORMALIZACION(INN)CHILE	5	4
Hong Kong, China	Hong Kong Accreditation Service	5	5
Chinese Taipei	TAF	2	1
Columbia	Organismo Nacional de Acreditacion de Colombia ONAC	1	1
Czech Republic	Czech Accreditation Institute	2	1
Cuba	ONARC(Organo Nacional de Acreditacin de la Repblica de Cuba)	2	0
Cyprus	ESYD(Hellenic Accreditation System S.A.)	1	1
Denmark	DANAK	2	2
Estonia	Estonian Accreditation Centre	1	1
Finland	FINAS Finnish Accreditation Service	2	2
Germany	Deutsche Akkreditierungsstelle GmbH	2	2
Guatemala	Oficina Guatemalteca de Acreditacin – OGA -	1	1
Greece	ESYD(Hellenic Accreditation System S.A.)	2	2
Honduras	Organismo Hondureo de Acreditacin/SEPLAN	2	0
Israel	Zahava Nizri	3	3
India	National Accreditation Board for Testing and Calibration Laboratories	4	4
Japan	Japan Accreditation Board (JAB)	2	2
Jordan	Jordan Standards and Metrology Organization-Accreditation Unit	2	0
Latvia	Latvian National Accreditation Bureau	1	1
Macedonia	Institute for Accreditation of Republic of Macedonia	1	1
Norway	Norwegian Accreditation	2	2
New Zealand	International Accreditation New Zealand(IANZ)	3	2
Portugal	Portuguese Accreditation Institute(IPAC)	2	2
Romania	RENAR – Romanian Accreditation Association	2	2
Sweden	SWEDAC	2	2
Switzerland	Swiss Accreditation Service	2	2
Slovenia	Slovenian Accreditation	2	2
Tunisia	Tunisian Accreditation Council TUNAC	1	1

Table A 2 Reported Results of Content of Lead

Lead (mg/kg)										
Lab Code	Sample No.		Testing Results (mg/kg)				Mean Result with Uncertainty (mg/kg)	Z-score	Limit of Quatitation (mg/kg)	Testing Method (Reagents ,Sample Treatment ,Operating Condition)
			1	2	3	4				
T074-01	F	635	0.67	0.67	0.68	0.66	0.67 ± 0.03	-4.17A	0.25	HCl-HNO ₃ ; Drying-Ashing; ICP
	G	634	0.73	0.83	0.78	0.78	0.78 ± 0.03	-4.38A	0.25	
T074-02	F	104	1.128	1.190	1.178	1.157	1.16 ± 0.16	-0.27	0.005	HNO ₃ -30%H ₂ O ₂ ; pressure digestion; AA
	G	733	1.025	0.940	0.955	0.855	0.94 ± 0.15	-3.27 A	0.005	
T074-03	F	101	1.04	1.16	1.14	1.07	1.10 ± 0.22	-0.75	0.005	HNO ₃ -HClO ₄ ; AA
	G	080	1.25	1.18	1.26	1.18	1.22 ± 0.24	-1.33	0.005	
T074-04	F	839	1.21	1.21	1.20	1.21	1.21 ± 0.01	0.13	0.00008	Microwave digestion; ICP-MS
	G	608	1.52	1.53	1.53	1.52	1.52 ± 0.01	0.76	0.00008	
T074-05	F	067	1.12	1.12	1.12	1.10	1.12 ± 0.18	-0.59	0.005	HNO ₃ Microwave digestion; AA
	G	801	1.20	1.21	1.24	1.22	1.22 ± 0.23	-1.33	0.005	
T074-06	F	098	1.31	1.27	1.24	1.24	1.27 ± 0.13	0.61	0.05	HNO ₃ -H ₂ O ₂ ; closed pressure digestion; ICP-MS
	G	793	1.57	1.48	1.50	1.45	1.50 ± 0.16	0.62	0.05	
T074-07	F	107	1.224	1.199	1.154	1.179	1.19 ± 0.03	-0.03	0.005	HNO ₃ ; AA
	G	193	1.410	1.389	1.371	1.358	1.38 ± 0.02	-0.21	0.005	
T074-08	F	119	1.11	1.14	1.17	1.19	1.15 ± 0.12	-0.35	0.03	
	G	598	1.34	1.34	1.38	1.31	1.34 ± 0.14	-0.49	0.03	
T074-09	F	563	<0.20	<0.20	<0.20	<0.20	<0.20		0.20	
	G	204	<0.20	<0.20	<0.20	<0.20	<0.20		0.20	
T074-10	F	838	0.56	0.56	0.56	0.56	0.56	-5.04A	0.05	AA
	G	648	0.99	0.99	0.99	0.99	0.99	-2.92W	0.05	
T074-11	F	437	1.23	1.02	1.28	1.28	1.21 ± 0.13	0.13	0.25	HNO ₃ -H ₂ O ₂ ; Microwave digestion; ICP
	G	133	1.34	1.57	1.47	1.47	1.46 ± 0.16	0.34	0.25	
T074-13	F	602	1.123	1.228	1.176	1.228	1.189 ± 0.02	-0.04	0.002	HNO ₃ -HPO ₄
	G	938	1.636	1.648	1.636	1.718	1.660 ± 0.02	1.73	0.002	
T074-14	F	605	1.45	1.46	1.30	1.42	1.41 ± 0.25	1.72	0.060	
	G	637	1.57	1.46	1.50	1.46	1.50 ± 0.27	0.62	0.060	
T074-15	F	493	1.14	1.17	—	—	1.16	-0.27	0.10	Open Vessel digestion; ICP-AE
	G	618	1.43	1.48	—	—	1.45	0.27	0.10	

Lead (mg/kg)										
Lab Code	Sample No.		Testing Results(mg/kg)				Mean Result with Uncertainty(mg/kg)	Z-score	Limit of Quatitation(mg/kg)	Method (Reagents ,Sample Treatment ,Operating Condition)
			1	2	3	4				
T074-16	F	596	1.22	1.22	1.16	1.16	1.19 ± 0.24	-0.03	0.20	HNO ₃ -H ₂ O ₂ ; AA
	G	699	1.42	1.42	1.40	1.40	1.41 ± 0.28	-0.01	0.20	
T074-17	F	781	1.10	1.09	1.20	1.22	1.15 ± 0.23	-0.35	0.02	HNO ₃ -H ₂ O ₂ ; Microwave digestion; ICP-MS
	G	643	1.46	1.57	1.37	1.59	1.50 ± 0.30	0.62	0.02	
T074-18	F	626	1.240	1.256	1.253	1.258	1.25 ± 0.31	0.45	0.03	HNO ₃ -H ₂ O ₂ ; Microwave digestion; ICP-MS
	G	262	1.545	1.565	1.540	1.534	1.55 ± 0.34	0.97	0.03	
T074-19	F	474	1.880	1.929	1.824	1.879	1.878 ± 0.150	5.44A	0.075	Microwave digestion; AA
	G	095	1.612	1.691	1.693	1.586	1.645 ± 0.132	1.63	0.075	
T074-20	F	621	0.900	0.988	1.022	0.929	0.960 ± 0.19	-1.86	0.1	HNO ₃ ; Dry
	G	138	1.364	1.391	1.024	1.125	1.226 ± 0.25	-1.28	0.1	
T074-26	F	399	—	—	—	—	—		—	
	G	084	—	—	—	—	—		—	
T074-27	F	534	1.50	1.45	1.48	1.48	1.48	2.28W	0.05	HNO ₃ -H ₂ O ₂ ; Microwave digestion; ICP-MS
	G	209	1.11	1.14	1.15	1.15	1.14	-1.88	0.05	
T074-28	F	615	1.28	1.23	1.22	1.29	1.26 ± 0.11	0.53	0.5	HNO ₃ ; Dry ashing; ICP-OES
	G	089	1.48	1.51	1.53	1.44	1.49 ± 0.13	0.55	0.5	
T074-31	F	641	1.16	1.19	1.21	1.23	1.20 ± 0.24	0.05	0.04	HNO ₃ -H ₂ O ₂ ; Close Vessel Microwave; ICP-SFMS
	G	210	1.45	1.44	1.44	1.49	1.46 ± 0.30	0.34	0.04	
T074-32	F	641	1.32	1.28	1.23	1.27	1.28 ± 0.26	0.68	0.02	HNO ₃ -H ₂ O ₂ ; Wet digestion in Microwave; ICP-MS
	G	935	1.50	1.36	1.43	1.34	1.41 ± 0.28	-0.01	0.02	
T074-33	F	581	1.07	1.13	1.22	1.31	1.18 ± 0.24	-0.11	0.05	HNO ₃ ; pressure digestion; ICP-OES
	G	090	1.47	1.48	1.36	1.47	1.45 ± 0.29	0.27	0.05	
T074-34	F	607	1.15	1.16	1.17	1.15	1.16 ± 0.23	-0.27	0.020	HNO ₃ -H ₂ O ₂ ; Microwave Closed Vessel; ICP-MS
	G	777	1.48	1.41	1.44	1.40	1.43 ± 0.29	0.13	0.020	
T074-35	F	590	1.22	1.20	1.20	1.17	1.19	-0.03	0.02	HNO ₃ -H ₂ O ₂ -H ₂ O; ICP-MS
	G	205	1.47	1.42	1.40	1.41	1.42	0.06	0.02	
T074-36	F	438	1.14	1.17	1.09	1.18	1.14 ± 0.22	-0.43	0.5	Acid Digestion
	G	611	1.35	1.38	1.32	1.41	1.36 ± 0.26	-0.35	0.5	

Lead (mg/kg)										
Lab Code	Sample No.		Testing Results(mg/kg)				Mean Result with Uncertainty(mg/kg)	Z-score	Limit of Quatitation(mg/kg)	Method (Reagents ,Sample Treatment ,Operating Condition)
			1	2	3	4				
T074-37	F	607	1.25	1.26	1.25	1.23	1.25 ± 0.13	0.45	0.10	HNO ₃ ; Microwave digestion ; ICP-MS
	G	700	1.50	1.47	1.45	1.51	1.48 ± 0.15	0.48	0.10	
T074-38	F	602	1.256	1.488	1.389	1.328	1.365 ± 0.14	1.36	0.05	HNO ₃ ; Ash Digestion ICP-MS
	G	616	1.556	1.586	1.485	1.495	1.531 ± 0.16	0.83	0.05	
T074-39	F	566	1.17	1.22	1.22	1.22	1.21 ± 0.25	0.13	0.1	HNO ₃ -H ₂ O ₂ ; high pressure Microwave ICP-MS
	G	208	1.40	1.33	1.35	1.48	1.39 ± 0.29	-0.14	0.1	
T074-40	F	632	1.39	1.36	1.31	1.34	1.35 ± 0.27	1.24	0.1	ICP-MS
	G	586	1.59	1.65	1.62	1.58	1.61 ± 0.32	1.38	0.1	
T074-41	F	788	1.92	1.78	1.76	—	1.82 ± 0.30	4.98A	0.012	HNO ₃ -H ₂ O ₂ ; wet digestion Microwave; AA
	G	132	2.05	2.23	2.19	—	2.16 ± 0.36	5.20A	0.012	
T074-42	F	063	1.085	1.066	1.091	1.186	1.107	-0.69	0.01	ICP-MS
	G	127	1.335	1.356	1.321	1.361	1.343	-0.47	0.01	
T074-43	F	619	1.913	1.865	1.892	1.879	1.89 ± 0.11	5.54A	0.01	HNO ₃ -HCl-H ₂ O ₂ ; AA
	G	592	1.592	1.569	1.560	1.586	1.58 ± 0.13	1.18	0.01	
T074-44	F	649	1.36	1.58	1.20	1.36	1.4 ± 0.2	1.64	0.070	HNO ₃ ; Acid Digestion ;ICP-MS
	G	174	1.12	1.20	1.44	1.45	1.3 ± 0.2	-0.77	0.07	
T074-45	F	532	0.74	0.69	0.78	0.86	0.77	-3.37A	0.01	AA
	G	088	0.97	1.00	1.13	1.30	1.10	-2.16W	0.01	
T074-46	F	587	1.24	1.11	1.13	1.13	1.15 ± 0.12	-0.35	0.1	HNO ₃ -H ₂ O; -
	G	583	1.44	1.39	1.36	1.39	1.40 ± 0.14	-0.08	0.1	
T074-47	F	116	1.19	1.22	1.20	1.21	1.20 ± 33%	0.05	0.006	ICP-MS
	G	741	1.47	1.45	1.45	1.47	1.46 ± 33%	0.34	0.006	
T074-49	F	638	1.210	1.270	1.290	1.210	1.245 ± 0.04	0.41	0.500	HNO ₃ -HCl; Microwave Closed Vessel; ICP-MS
	G	324	1.470	1.430	1.450	1.480	1.458 ± 0.02	0.33	0.5	
T074-50	F	609	1.25	1.24	1.26	1.25	1.25 ± 28.2%	0.45	0.004	HNO ₃ -H ₂ O; Microwave ICP-MS
	G	129	1.52	1.55	1.54	1.50	1.54 ± 28.2%	0.90	0.004	
T074-52	F	646	0.94	1.15	1.06	1.10	1.06 ± 0.23	-1.07	0.08	HNO ₃ -HCl; Dry-Ashing; AA
	G	325	1.23	1.23	1.09	1.24	1.20 ± 0.26	-1.46	0.08	

Lead (mg/kg)										
Lab Code	Sample No.		Testing Results(mg/kg)				Mean Result with Uncertainty(mg/kg)	Z-score	Limit of Quatitation(mg/kg)	Method (Reagents ,Sample Treatment ,Operating Condition)
			1	2	3	4				
T074-53	F	776	1.17	1.16	1.16	—	1.163 ± 0.158	-0.25	0.0199	HNO ₃ -HCl; Dry-Ashing; AAS
	G	894	1.38	1.30	1.30	—	1.327 ± 0.180	-0.58	0.0507	
T074-54	F	131	1.01	1.05	1.06	1.05	1.04 ± 0.06	-1.22	0.20	HNO ₃ -Mg(NO ₃) ₂ -NH ₄ H ₂ PO ₄
	G	827	1.32	1.24	1.24	1.29	1.27 ± 0.07	-0.98	0.35	
T074-55	F	113	1.31	1.24	1.31	1.36	1.31 ± 0.1	0.92	0.10	Acid Digestion ICP-OES
	G	317	1.54	1.54	1.53	1.46	1.52 ± 0.1	0.76	0.10	
T074-56	F	430	1.15	1.20	1.09	1.14	1.15 ± 0.11	-0.35	0.10	Dry-ashing AAS
	G	332	1.41	1.45	1.46	1.43	1.44 ± 0.11	0.20	0.10	
T074-57	F	737	1.10	1.01	0.82	0.87	0.95 ± 0.15	-1.94	0.10	HNO ₃ -H ₂ O; ICP-OES
	G	313	1.21	1.30	1.14	1.40	1.26 ± 0.15	-1.05	0.10	
T074-58	F	454	1.18	1.16	1.17	1.15	1.17 ± 0.03	-0.19	0.011	HNO ₃ ; Microwave ICP-MS
	G	278	1.39	1.41	1.40	1.39	1.40 ± 0.02	-0.08	0.011	
T074-59	F	770	1.13	1.15	1.19	1.17	1.16	-0.27	0.05	HNO ₃ -HCl-H ₂ O ₂ ;
	G	329	1.45	1.46	1.43	1.37	1.43	0.13	0.05	
T074-61	F	445	0.99	1.01	0.99	1.03	1.01	-1.46	0.01	HNO ₃ -Mg(NO ₃) ₂ -NH ₄ H ₂ PO ₄ AA
	G	739	1.23	1.21	1.27	1.28	1.25	-1.12	0.05	
T074-62	F	462	1.44	1.39	1.40	1.34	1.39 ± 0.05	1.56	0.08	HNO ₃ -H ₂ O Microwave Digestion
	G	767	1.62	1.63	1.54	1.57	1.59 ± 0.05	1.24	0.08	
T074-63	F	110	1.163	1.233	1.183	1.198	1.19 ± 0.24	-0.03	0.02	HNO ₃ -H ₂ O ₂ ; Acid Digestion ;ICP-MS
	G	829	1.586	1.584	1.375	1.522	1.52 ± 0.30	0.76	0.02	
T074-64	F	441	0.99	0.98	1.07	1.11	1.04 ± 0.29	-1.22	0.10	HNO ₃ -H ₂ O ₂ ; Microwave Digestion
	G	041	1.19	1.26	1.27	1.36	1.27 ± 0.36	-0.98	0.10	
T074-65	F	766	0.79	0.89	0.95	0.87	0.88 ± 0.26	-2.50W	0.016	HNO ₃ -H ₂ O ₂ -Mg(NO ₃) ₂ -NH ₄ H ₂ PO ₄ ; Microwave Digestion; AA
	G	304	1.08	1.05	1.05	1.23	1.10 ± 0.33	-2.16W	0.016	
T074-66	F	456	1.24	1.37	1.30	1.34	1.31 ± 0.23	0.92	0.025	HNO ₃ -H ₂ O ₂ ; Acid Digestion
	G	321	1.55	1.47	1.49	1.54	1.51 ± 0.23	0.69	0.025	
T074-67	F	434	1.14	1.08	1.02	1.07	1.08 ± 0.16	-0.91	0.020	HNO ₃ ; Microwave Closed Vessel; ICP-OES
	G	328	1.33	1.35	1.35	1.36	1.35 ± 0.20	-0.42	0.020	

Lead (mg/kg)										
LabCode	Sample No.		Testing Results (mg/kg)				Mean Result with Uncertainty(mg/kg)	Z-score	Limit of Quatitation(mg/kg)	Method (Reagents ,Sample Treatment ,Operating Condition)
			1	2	3	4				
T074-68	F	435	1.19	1.17	1.19	1.18	1.18 ± 0.1	-0.11	0.01	HNO ₃ ; Microwave ICP-MS
	G	731	1.44	1.41	1.43	1.42	1.42 ± 0.2	0.06	0.01	
T074-69	F	654	1.20	1.24	1.20	1.21	1.21 ± 0.27	0.13	0.010	HNO ₃ ; Microwave ICP-MS
	G	633	1.52	1.47	1.42	1.43	1.46 ± 0.32	0.34	0.010	
T074-70	F	464	1.18	1.26	1.26	1.31	1.25 , U=0.17	0.45	0.05	HNO ₃ ; Microwave ICP-MS
	G	457	1.56	1.42	1.51	1.45	1.49, U=0.20	0.55	0.05	
T074-71	F	453	1.12	1.09	1.13	1.13	1.12 ± 0.034	-0.59	0.01	HNO ₃ -H ₂ O ₂ ; Acid Digestion ;ICP-MS
	G	284	1.38	1.35	1.35	1.36	1.36 ± 0.027	-0.35	0.01	
T074-72	F	432	1.22	1.21	1.21	1.24	1.22 ± 0.19	1.00	0.05	HNO ₃ -HCl; ICP-MS
	G	743	1.44	1.52	1.57	1.56	1.52 ± 0.24	0.76	0.05	

Note1: The value marked with “A”, which is $|z| \geq 3.0$; the value marked with “W”, which is $2.0 < |z| < 3.0$.

Note2: The test results of Lab T074-10 have not been included in this statistical analysis.

Table A 3 Statistics for the Test on Contents of Lead

	Result F	Result G
Total Number of Results	59	59
Robust Mean, mg/kg	1.194	1.411
Robust Standard Deviation, mg/kg	0.126	0.144
Standard uncertainty	0.021	0.024
Minimum, mg/kg	0.67	0.78
Maximum, mg/kg	1.89	2.16
Range, mg/kg	1.22	1.38

Table A 4 Reported Results of Content of Cadmium

Cadmium (mg/kg)										
Lab Code	Sample No.		Testing Results(mg/kg)				Mean Result with Uncertainty(mg/kg)	Z-score	Limit of Quatitation(mg /kg)	Method (Reagents ,Sample Treatment ,Operating Condition)
			1	2	3	4				
T074-01	F	635	1.14	1.16	1.15	1.16	1.15 ± 0.04	-1.46	0.003	HCl-HNO ₃ ; Drying-Ashing; ICP
	G	634	0.90	0.89	0.97	0.93	0.91 ± 0.04	-1.41	0.003	
T074-02	F	104	1.438	1.455	1.423	1.400	1.43 ± 0.18	0.34	0.0001	HNO ₃ -30%H ₂ O ₂ ; pressure digestion; AA
	G	733	1.257	1.211	1.288	1.244	1.25 ± 0.15	0.90	0.0001	
T074-03	F	101	1.45	1.43	1.34	1.45	1.42 ± 0.28	0.28	0.0001	HNO ₃ -HClO ₄ ; AA
	G	080	1.11	1.20	1.13	1.15	1.15 ± 0.23	0.22	0.0001	
T074-04	F	839	0.99	1.00	1.00	0.99	1.00 ± 0.01	-2.43W	0.00003	Microwave digestion; ICP-MS
	G	608	0.74	0.74	0.74	0.75	0.74 ± 0.01	-2.57W	0.00003	
T074-05	F	067	1.42	1.41	1.41	1.41	1.41 ± 0.22	0.21	0.001	HNO ₃ Microwave digestion; AA
	G	801	1.21	1.20	1.20	1.21	1.20 ± 0.19	0.56	0.001	
T074-06	F	098	1.29	1.29	1.35	1.31	1.31 ± 0.12	-0.43	0.01	HNO ₃ -H ₂ O ₂ ; closed pressure digestion; ICP-MS
	G	793	1.02	1.12	1.02	1.06	1.06 ± 0.13	-0.39	0.01	
T074-07	F	107	1.422	1.425	1.443	1.415	1.43 ± 0.01	0.34	0.005	HNO ₃ ; AA
	G	193	1.172	1.146	1.118	1.144	1.15 ± 0.02	0.22	0.005	
T074-08	F	119	1.50	1.47	1.49	1.46	1.48 ± 0.16	0.67	0.04	
	G	598	1.12	1.16	1.16	1.14	1.15 ± 0.13	0.22	0.04	
T074-09	F	563	1.23	1.19	1.23	1.23	1.22	-1.01	0.05	
	G	204	0.94	0.98	0.99	0.99	0.98	-0.94	0.05	
T074-10	F	838	1.39	1.45	1.33	1.18	1.38	0.02	0.05	AA
	G	648	1.23	1.15	1.17	1.26	1.18	0.42	0.05	
T074-11	F	437	1.61	1.58	1.60	1.60	1.60 ± 0.26	1.44	0.05	HNO ₃ -30%H ₂ O ₂ ; Microwave digestion; ICP
	G	133	1.17	1.24	1.25	1.30	1.24 ± 0.20	0.83	0.05	
T074-13	F	602	1.792	1.772	—	—	1.782 ± 0.025	2.62W	0.0005	HNO ₃ -HPO ₄
	G	938	1.354	1.382	—	—	1.368 ± 0.025	1.70	0.0005	
T074-14	F	605	1.38	1.28	1.23	1.37	1.32 ± 0.26	-0.37	0.008	
	G	637	1.00	0.92	0.95	0.94	0.95 ± 0.19	-1.14	0.008	
T074-15	F	493	1.29	1.29	—	—	1.29	-0.56	0.03	Open Vessel digestion; ICP-AE
	G	618	1.00	1.05	—	—	1.025	-0.63	0.03	

Cadmium (mg/kg)										
LabCode	Sample No.		Testing Results(mg/kg)				Mean Result with Uncertainty(mg/kg)	Z-score	Limit of Quatitation(mg/kg)	Method (Reagents ,Sample Treatment ,Operating Condition)
			1	2	3	4				
T074-16	F	596	1.64	1.64	1.79	1.79	1.71 ± 0.14	2.15W	0.03	HNO ₃ -H ₂ O ₂ ; AA
	G	699	1.17	1.17	1.32	1.32	1.24 ± 0.10	0.83	0.03	
T074-17	F	781	1.49	1.57	1.37	1.50	1.48 ± 0.30	0.67	0.03	HNO ₃ -H ₂ O ₂ ; Microwave digestion; ICP-MS
	G	643	1.19	1.16	1.19	1.23	1.19 ± 0.23	0.49	0.03	
T074-18	F	626	1.528	1.526	1.540	1.536	1.53 ± 0.31	0.99	0.005	HNO ₃ -H ₂ O ₂ ; Microwave digestion; ICP-MS
	G	262	1.235	1.247	1.241	1.233	1.24 ± 0.25	0.83	0.005	
T074-19	F	474	1.336	1.377	1.330	1.269	1.328 ± 0.079	-0.31	0.005	Microwave digestion; AA
	G	095	1.002	1.019	1.084	1.088	1.048 ± 0.094	-0.47	0.005	
T074-20	F	621	0.986	0.991	0.967	0.952	0.974 ± 0.20	-2.60W	0.01	HNO ₃ ; Dry
	G	138	0.762	0.767	0.763	0.751	0.761 ± 0.15	-2.43W	0.01	
T074-26	F	399	1.07	0.958	1.04	1.11	1.04 ± 0.04	-2.17W	0.01	HNO ₃ ; ICP
	G	084	0.777	0.745	0.803	0.824	0.79 ± 0.04	-2.23W	0.01	
T074-27	F	534	1.19	1.16	1.18	1.17	1.18	-1.27	0.05	HNO ₃ -H ₂ O ₂ ; Microwave digestion; ICP-MS
	G	209	1.37	1.40	1.42	1.41	1.40	1.92	0.05	
T074-28	F	615	1.46	1.47	1.45	1.46	1.46 ± 0.03	0.54	0.05	HNO ₃ ; Dry ashing; ICP-OES
	G	089	1.17	1.16	1.16	1.16	1.16 ± 0.03	0.29	0.05	
T074-31	F	641	1.47	1.54	1.57	1.55	1.53 ± 0.29	0.99	0.005	HNO ₃ -H ₂ O ₂ ; Close Vessel Microwave; ICP-SFMS
	G	210	1.22	1.21	1.23	1.27	1.23 ± 0.24	0.77	0.005	
T074-32	F	641	1.46	1.40	1.38	1.42	1.42 ± 0.28	0.28	0.01	HNO ₃ -H ₂ O ₂ ; Wet digestion in Microwave; ICP-MS
	G	935	1.09	0.98	1.06	1.00	1.03 ± 0.21	-0.60	0.01	
T074-33	F	581	1.42	1.43	1.41	1.42	1.42 ± 0.21	0.28	0.02	HNO ₃ ; pressure digestion; ICP-OES
	G	090	1.13	1.12	1.12	1.13	1.13 ± 0.17	0.08	0.02	
T074-34	F	607	1.36	1.36	1.35	1.36	1.36 ± 0.19	-0.11	0.010	HNO ₃ -H ₂ O ₂ ; Microwave Closed Vessel; ICP-MS
	G	777	1.09	1.10	1.07	1.05	1.08 ± 0.15	-0.26	0.010	
T074-35	F	590	1.48	1.41	1.48	1.48	1.46	0.54	0.001	HNO ₃ -H ₂ O ₂ ; ICP-MS
	G	205	1.18	1.13	1.16	1.15	1.15	0.22	0.001	
T074-36	F	438	1.43	1.50	1.44	1.46	1.46 ± 0.38	0.54	0.025	Acid Digestion
	G	611	1.12	1.16	1.13	1.33	1.18 ± 0.31	0.42	0.025	

Cadmium (mg/kg)										
LabCode	Sample No.		Testing Results(mg/kg)				Mean Result with Uncertainty(mg/kg)	Z-score	Limit of Quatitation(mg/kg)	Method (Reagents ,Sample Treatment ,Operating Condition)
			1	2	3	4				
T074-37	F	607	1.52	1.46	1.46	1.54	1.50 ± 0.15	0.80	0.05	HNO ₃ ; Microwave digestion ; ICP-MS
	G	700	1.17	1.17	1.17	1.15	1.17 ± 0.12	0.36	0.05	
T074-38	F	602	1.473	1.550	1.527	1.451	1.500 ± 0.12	0.80	0.02	HNO ₃ ; Ash Digestion ICP-MS
	G	616	1.224	1.173	1.120	1.133	1.163 ± 0.09	0.31	0.02	
T074-39	F	566	1.38	1.31	1.29	1.37	1.34 ± 0.19	-0.24	0.02	HNO ₃ -H ₂ O ₂ ; high pressure Microwave ICP-MS
	G	208	1.09	0.94	0.99	0.91	0.98 ± 0.14	-0.94	0.02	
T074-40	F	586	1.13	1.18	1.17	1.20	1.17 ± 0.23	-1.33	0.02	ICP-MS
	G	632	1.49	1.50	1.49	1.46	1.48 ± 0.30	2.47W	0.02	
T074-41	F	788	1.55	1.73	1.85	—	1.71 ± 0.29	2.15W	0.001	HNO ₃ -H ₂ O ₂ ; wet digestion Microwave; AA
	G	132	1.34	1.04	1.61	—	1.33 ± 0.22	1.45	0.001	
T074-42	F	063	1.362	1.290	1.322	1.371	1.336	-0.26	0.01	ICP-MS
	G	127	1.068	1.038	1.098	1.073	1.069	-0.33	0.01	
T074-43	F	619	1.243	1.305	1.33	1.207	1.27 ± 0.18	-0.69	0.001	HNO ₃ -HCl-H ₂ O ₂ ; AA
	G	592	1.694	1.705	1.635	1.518	1.64 ± 0.27	3.56A	0.001	
T074-44	F	649	1.11	1.27	1.43	1.63	1.4 ± 0.2	0.15	0.040	HNO ₃ ; Acid Digestion ;ICP-MS
	G	174	1.40	1.49	1.13	1.14	1.3 ± 0.2	1.24	0.040	
T074-45	F	532	0.70	0.75	0.73	0.81	0.75	-4.05A	0.01	AA
	G	088	0.54	0.62	0.74	0.75	0.66	-3.11A	0.01	
T074-46	F	587	1.40	1.36	1.34	1.35	1.36 ± 0.14	-0.11	0.04	HNO ₃ -H ₂ O; -
	G	583	1.19	1.11	1.00	1.10	1.10 ± 0.11	-0.12	0.04	
T074-47	F	116	1.45	1.47	1.44	1.45	1.45 ± 16%	0.47	0.0007	ICP-MS
	G	741	1.15	1.14	1.15	1.17	1.15 ± 16%	0.22	0.0007	
T074-49	F	638	1.450	1.490	1.490	1.510	1.485 ± 0.03	0.70	0.100	HNO ₃ -HCl; Microwave Closed Vessel; ICP-MS
	G	324	1.130	1.170	1.170	1.170	1.160 ± 0.005	0.29	0.100	
T074-50	F	609	1.48	1.48	1.48	1.48	1.48 ± 38.2%	0.67	0.001	HNO ₃ -H ₂ O; Microwave ICP-MS
	G	129	1.15	1.16	1.16	1.14	1.16 ± 38.2%	0.29	0.001	
T074-52	F	646	1.01	1.02	1.00	1.04	1.02 ± 0.18	-2.30W	0.02	HNO ₃ -HCl; Dry-Ashing; AA
	G	325	0.76	0.82	0.79	0.82	0.80 ± 0.14	-2.16W	0.022	

Cadmium (mg/kg)										
LabCode	Sample No.		Testing Results(mg/kg)				Mean Result with Uncertainty(mg/kg)	Z-score	Limit of Quatitation(mg/kg)	Method (Reagents ,Sample Treatment ,Operating Condition)
			1	2	3	4				
T074-53	F	776	1.03	1.07	1.05	—	1.05 ± 0.10	-2.11W	0.0085	HNO ₃ -HCl; Dry-Ashing; AAS
	G	894	0.79	0.79	0.79	—	0.79 ± 0.079	-2.23W	0.023	
T074-54	F	131	1.44	1.37	1.42	1.41	1.41 ± 0.060	0.21	0.26	HNO ₃ -Mg(NO ₃) ₂ -NH ₄ H ₂ PO ₄
	G	827	1.09	1.09	1.09	1.09	1.09 ± 0.05	-0.19	0.01	
T074-55	F	113	1.38	1.40	1.33	1.39	1.38 ± 0.1	0.02	0.05	Acid Digestion ICP-OES
	G	317	1.04	1.11	1.24	1.17	1.11 ± 0.1	-0.05	0.05	
T074-56	F	430	1.28	1.36	1.43	1.44	1.38 ± 0.09	0.02	0.01	Dry-ashing AAS
	G	332	1.33	1.34	1.35	1.31	1.33 ± 0.09	1.45	0.01	
T074-57	F	737	1.20	1.15	1.32	1.01	1.17 ± 0.12	-1.33	0.05	HNO ₃ -H ₂ O; ICP-OES
	G	313	0.92	0.78	1.03	0.85	0.90 ± 0.12	-1.48	0.05	
T074-58	F	454	1.40	1.39	1.40	1.38	1.39 ± 0.02	0.09	0.0047	HNO ₃ ; Microwave ICP-MS
	G	278	1.09	1.11	1.09	1.09	1.10 ± 0.02	-0.12	0.0047	
T074-59	F	770	1.36	1.38	1.43	1.42	1.40	0.15	0.01	HNO ₃ -HCl-H ₂ O ₂ ;
	G	329	1.14	1.13	1.17	1.14	1.15	0.22	0.01	
T074-61	F	445	1.27	1.23	1.28	1.26	1.26	-0.75	0.01	HNO ₃ -Mg(NO ₃) ₂ -NH ₄ H ₂ PO ₄ AA
	G	739	1.06	1.05	1.03	1.04	1.04	-0.53	0.05	
T074-62	F	462	1.37	1.42	1.41	1.39	1.39 ± 0.02	0.09	0.0015	HNO ₃ -H ₂ O Microwave Digestion
	G	767	1.13	1.13	1.14	1.13	1.13 ± 0.005	0.08	0.0015	
T074-63	F	110	1.454	1.480	1.454	1.442	1.46 ± 0.29	0.54	0.02	HNO ₃ -H ₂ O ₂ ; Acid Digestion ;ICP-MS
	G	829	1.240	1.268	1.126	1.151	1.20 ± 0.24	0.56	0.02	
T074-64	F	441	1.56	1.39	1.59	1.57	1.53 ± 0.38	0.99	0.02	HNO ₃ -H ₂ O ₂ ; Microwave Digestion
	G	041	1.22	1.26	1.17	1.27	1.23 ± 0.30	0.77	0.02	
T074-65	F	766	1.49	1.43	1.38	1.35	1.41 ± 0.45	0.21	0.010	HNO ₃ -H ₂ O ₂ -Mg(NO ₃) ₂ - NH ₄ H ₂ PO ₄ ; Microwave Digestion; AA
	G	304	1.10	1.14	1.02	1.23	1.12 ± 0.36	0.02	0.010	
T074-66	F	456	1.56	1.81	1.64	1.79	1.70 ± 0.26	2.09W	0.025	HNO ₃ -H ₂ O ₂ ; Acid Digestion
	G	321	1.08	1.03	1.07	1.19	1.09 ± 0.16	-0.19	0.025	
T074-67	F	434	1.39	1.35	1.32	1.37	1.36 ± 0.13	-0.11	0.005	HNO ₃ ; Microwave Closed Vessel; ICP-OES
	G	328	1.07	1.08	1.11	1.11	1.09 ± 0.11	-0.19	0.005	

Cadmium (mg/kg)										
LabCode	Sample No.		Testing Results(mg/kg)				Mean Result with Uncertainty(mg/kg)	Z-score	Limit of Quatitation(mg/kg)	Method (Reagents ,Sample Treatment ,Operating Condition)
			1	2	3	4				
T074-68	F	435	1.45	1.45	1.45	1.45	1.45 ± 0.2	0.47	0.01	HNO ₃ ; Microwave ICP-MS
	G	731	1.15	1.15	1.14	1.15	1.15 ± 0.1	0.22	0.01	
T074-69	F	654	1.11	1.12	1.10	1.13	1.12 ± 0.20	-1.66	0.010	HNO ₃ ; Microwave ICP-MS
	G	633	0.91	0.90	0.88	0.88	0.89 ± 0.16	-1.55	0.010	
T074-70	F	464	1.41	1.39	1.44	1.40	1.41, U=0.12	0.21	0.01	HNO ₃ ; Microwave ICP-MS
	G	457	1.14	1.08	1.08	1.09	1.10, U=0.10	-0.12	0.01	
T074-71	F	453	1.34	1.29	1.32	1.33	1.32 ± 0.040	-0.37	0.002	HNO ₃ -H ₂ O ₂ ; Acid Digestion ;ICP-MS
	G	284	1.12	1.07	1.05	1.05	1.07 ± 0.067	-0.32	0.002	
T074-72	F	432	1.40	1.42	1.44	1.57	1.46 ± 0.19	0.54	0.02	HNO ₃ -HCl; ICP-MS
	G	743	1.11	1.12	1.27	1.26	1.20 ± 0.16	0.56	0.02	

Note1: The value marked with “A”, which is $|z| \geq 3.0$; the value marked with “W”, which is $2.0 < |z| < 3.0$.

Note2: The test results of Lab T074-10 have not be included in this statistical analysis.

Table A 5 Statistics for the Test on Contents of Cadmium

	Result F	Result G
Total Number of Results	61	61
Robust Mean, mg/kg	1.377	1.118
Robust Standard Deviation, mg/kg	0.155	0.147
Standard uncertainty	0.025	0.024
Minimum, mg/kg	0.75	0.66
Maximum, mg/kg	1.782	1.64
Range, mg/kg	1.032	0.98

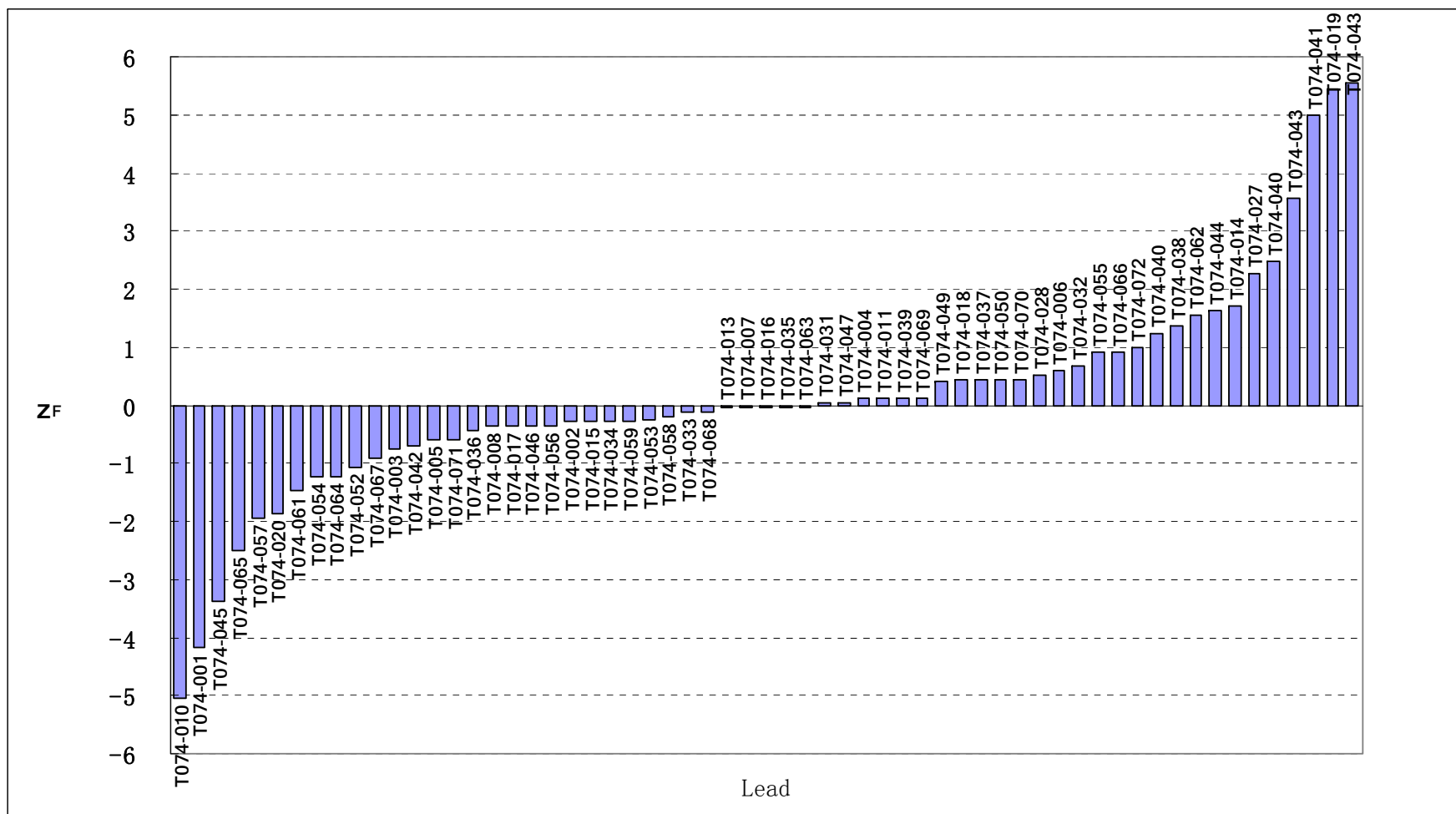
APPENDIX B

Graphical Display of Testing Results

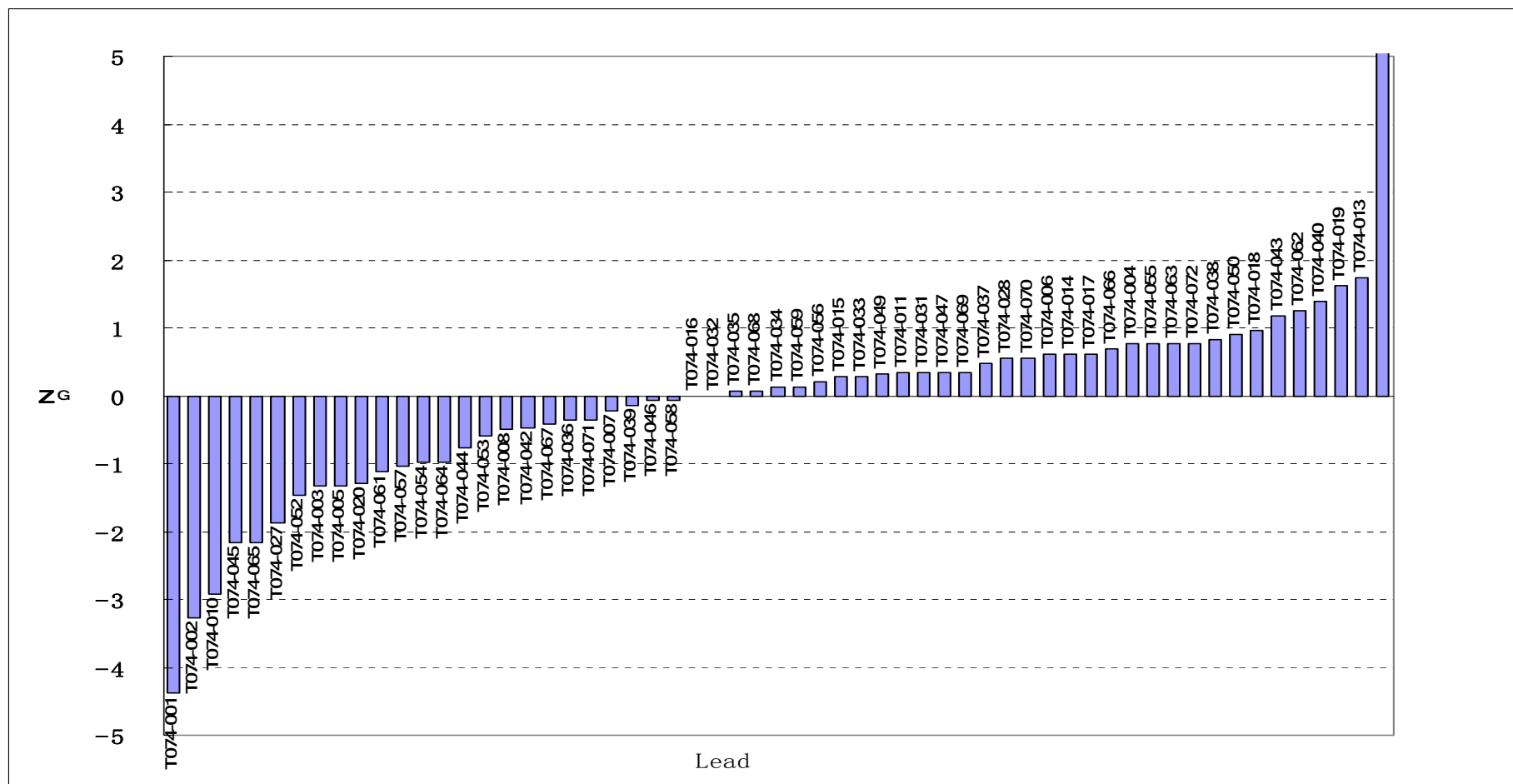
Here, graphs were made in order to give a clear display of testing results and their statistics.

Ordered z-score bar charts (B.1~B.4) are given in order to facilitate visual identification of the warning and action results. Histograms of z-scores (B.5~B.8) show the distributions of testing results. Youden Plots of paired z-scores (B.9~B.10) distinctly reveal questionable and unsatisfactory results. Points outside the ellipses indicate their well-separated from the rest of the data, where points far out along the major axis of the ellipses imply that the results are subject to bias or large variation, and those points far away from the major axis represent participants whose repeatability is poor. Generally, labs with large absolute between-labs z-scores should be outside the ellipse in quadrants 2 and 4, while labs with large absolute within-lab z-scores should be outside the ellipse in quadrants 1 and 3. Some points are too far away from the ellipses to be drawn on the Youden Plots, the corresponding lab codes are drawn together with arrows that indicate their orientations.

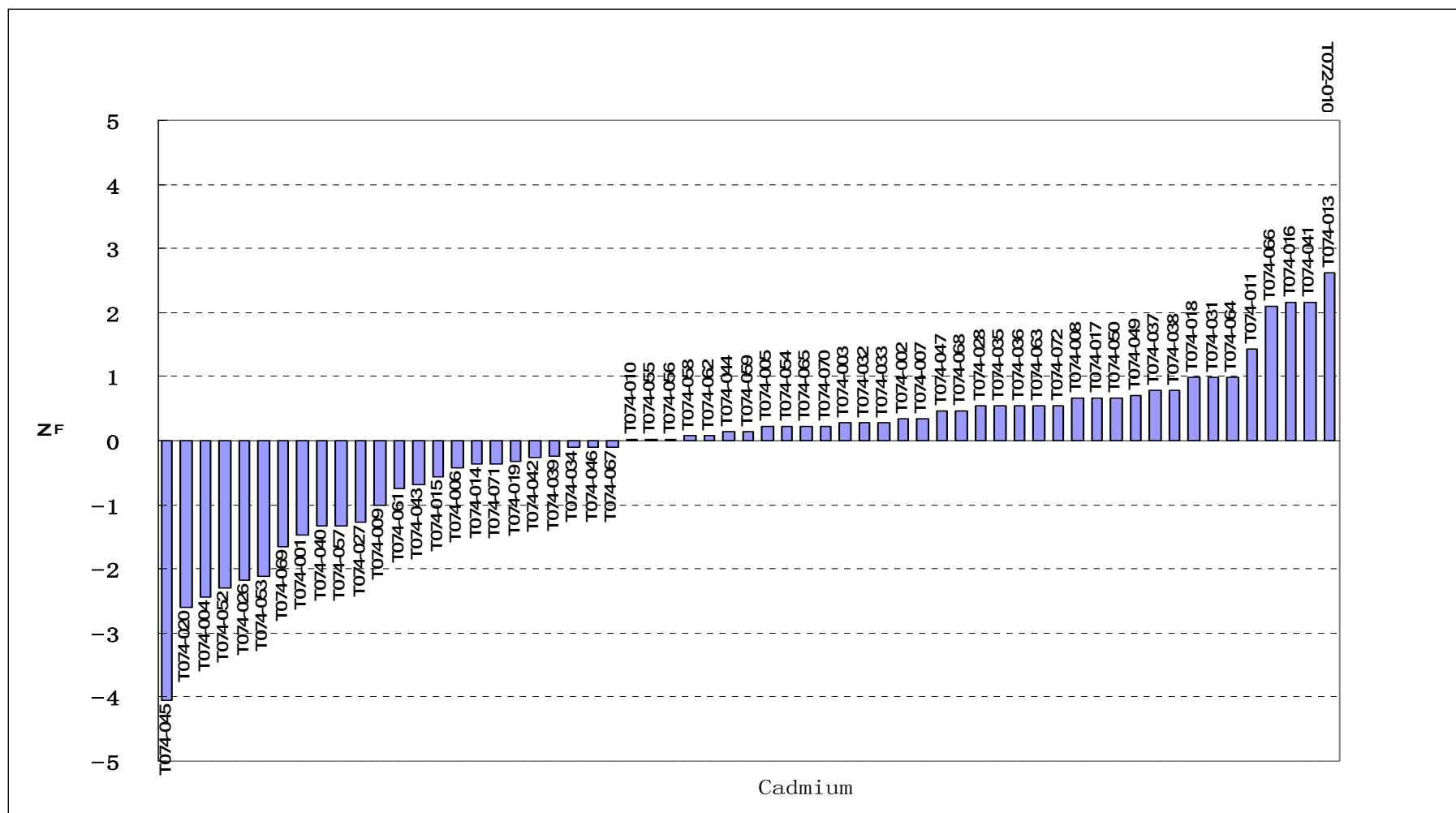
B.1 Ordered z-scores Bar Chart of Testing Results on Lead Content in Sample F



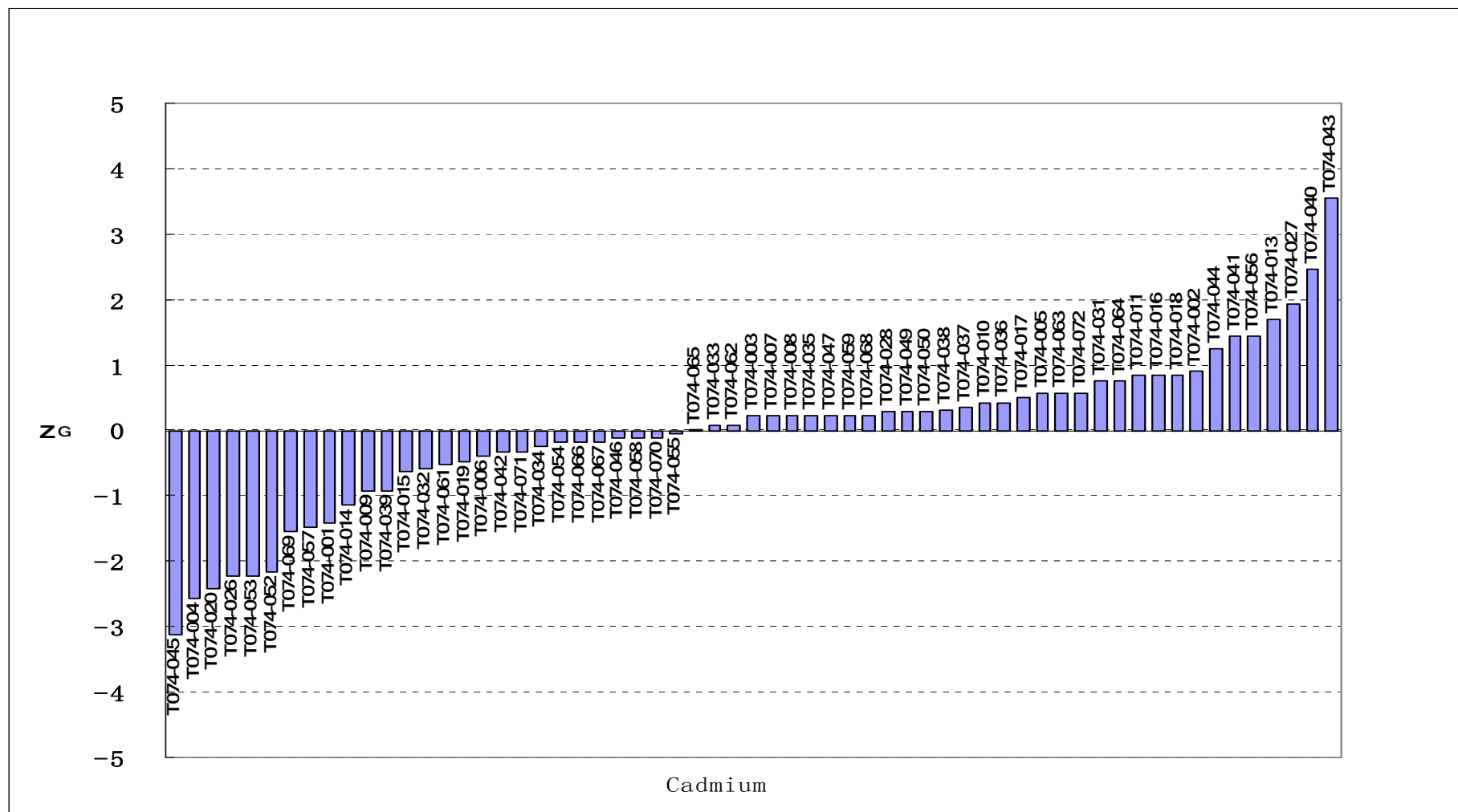
B.2 Ordered z-scores Bar Chart of Testing Results on Lead Content in Sample G



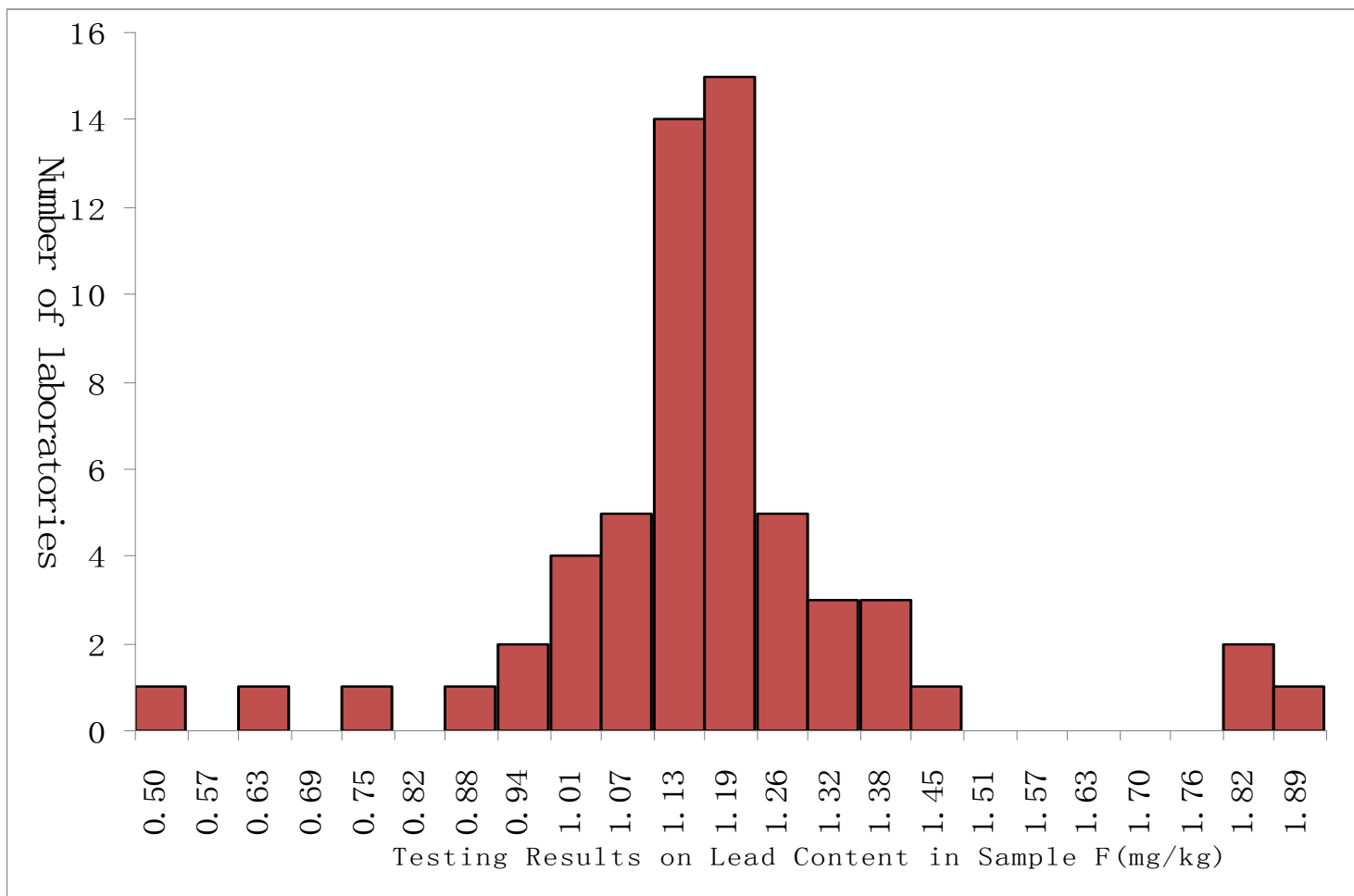
B.3 Ordered z-scores Bar Chart of Testing Results on Cadmium Content in Sample F



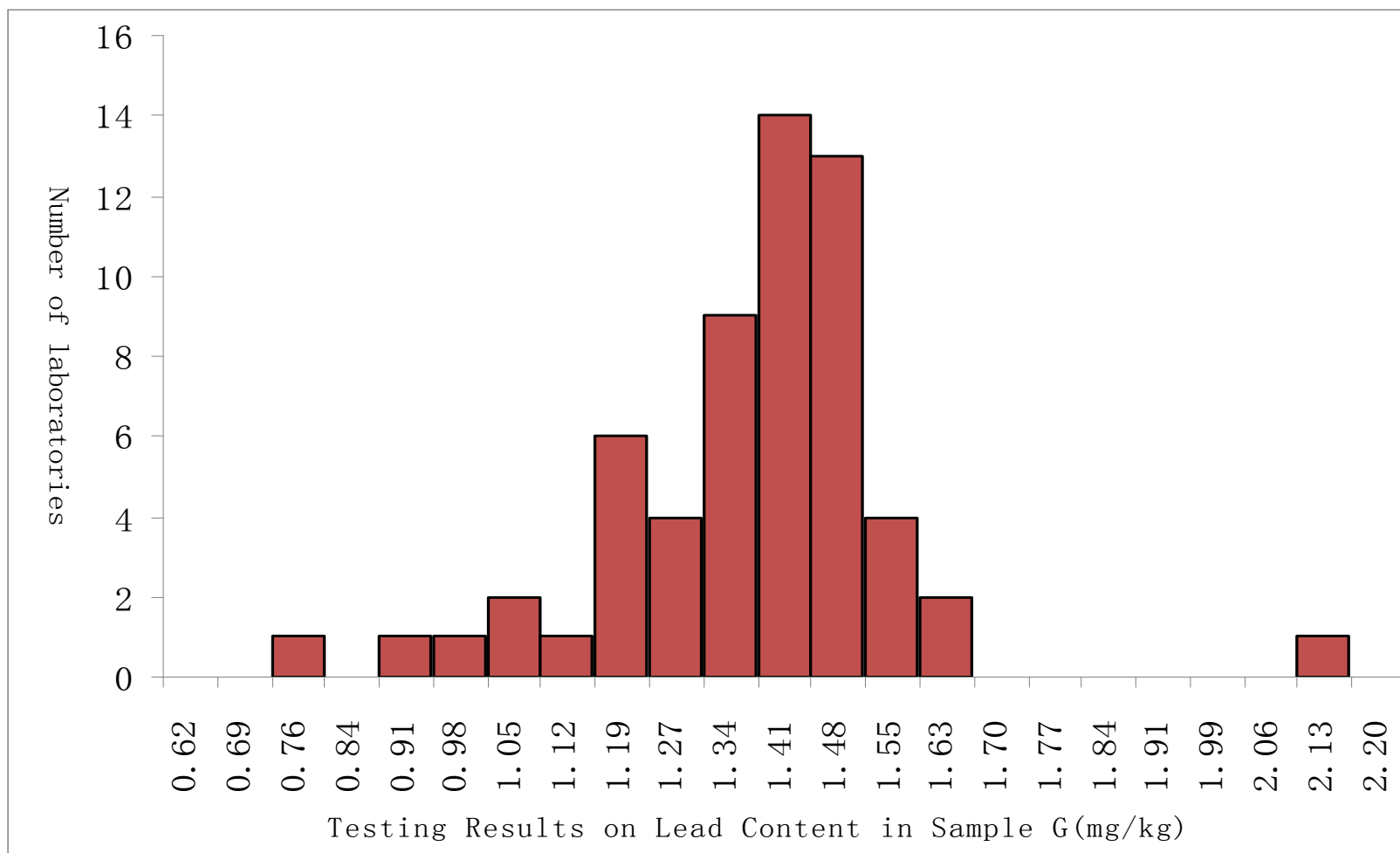
B.4 Ordered z-scores Bar Chart of Testing Results on Cadmium Content in Sample G



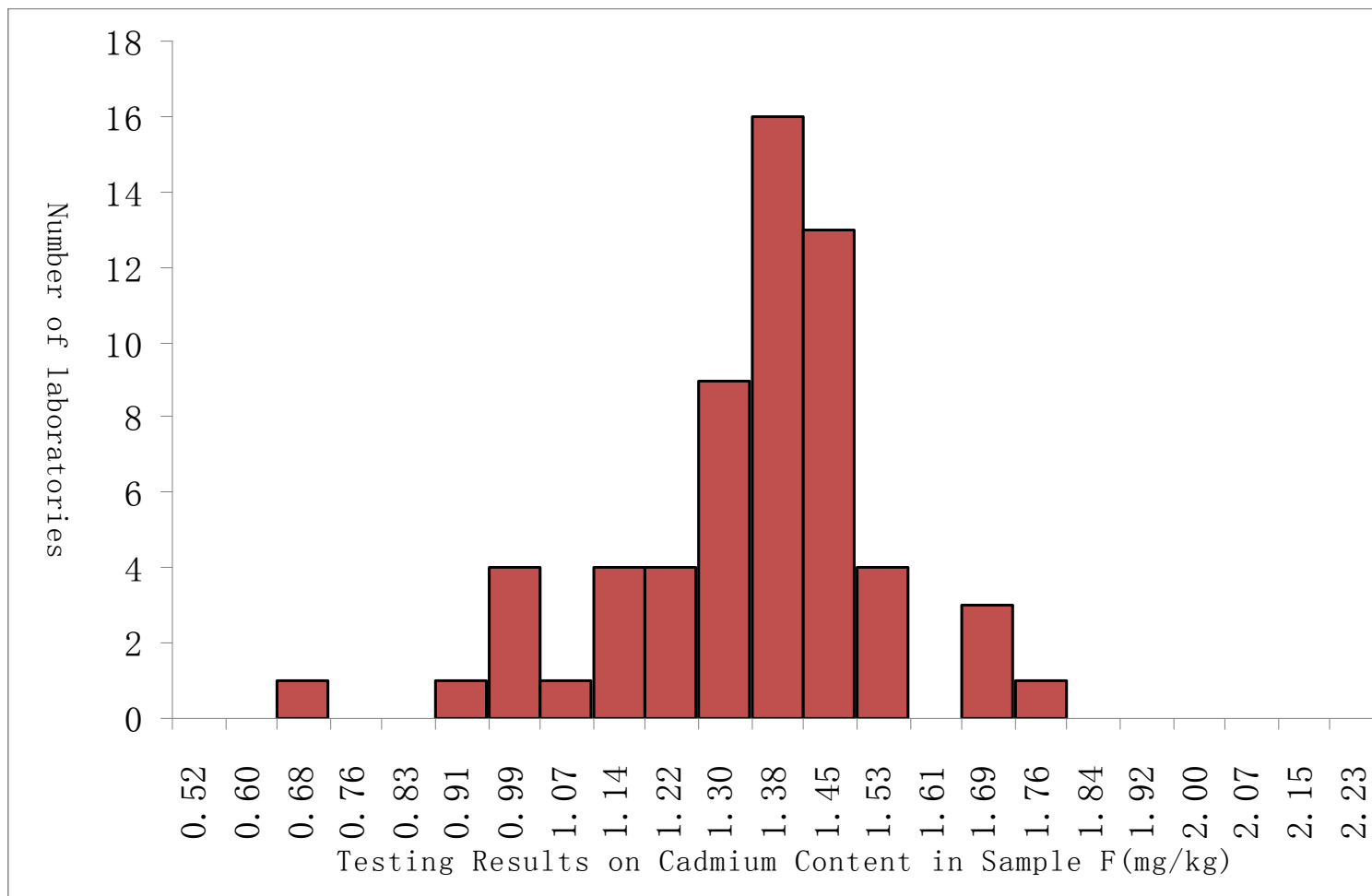
B.5 Distribution of Testing Results on Lead Content in Sample F



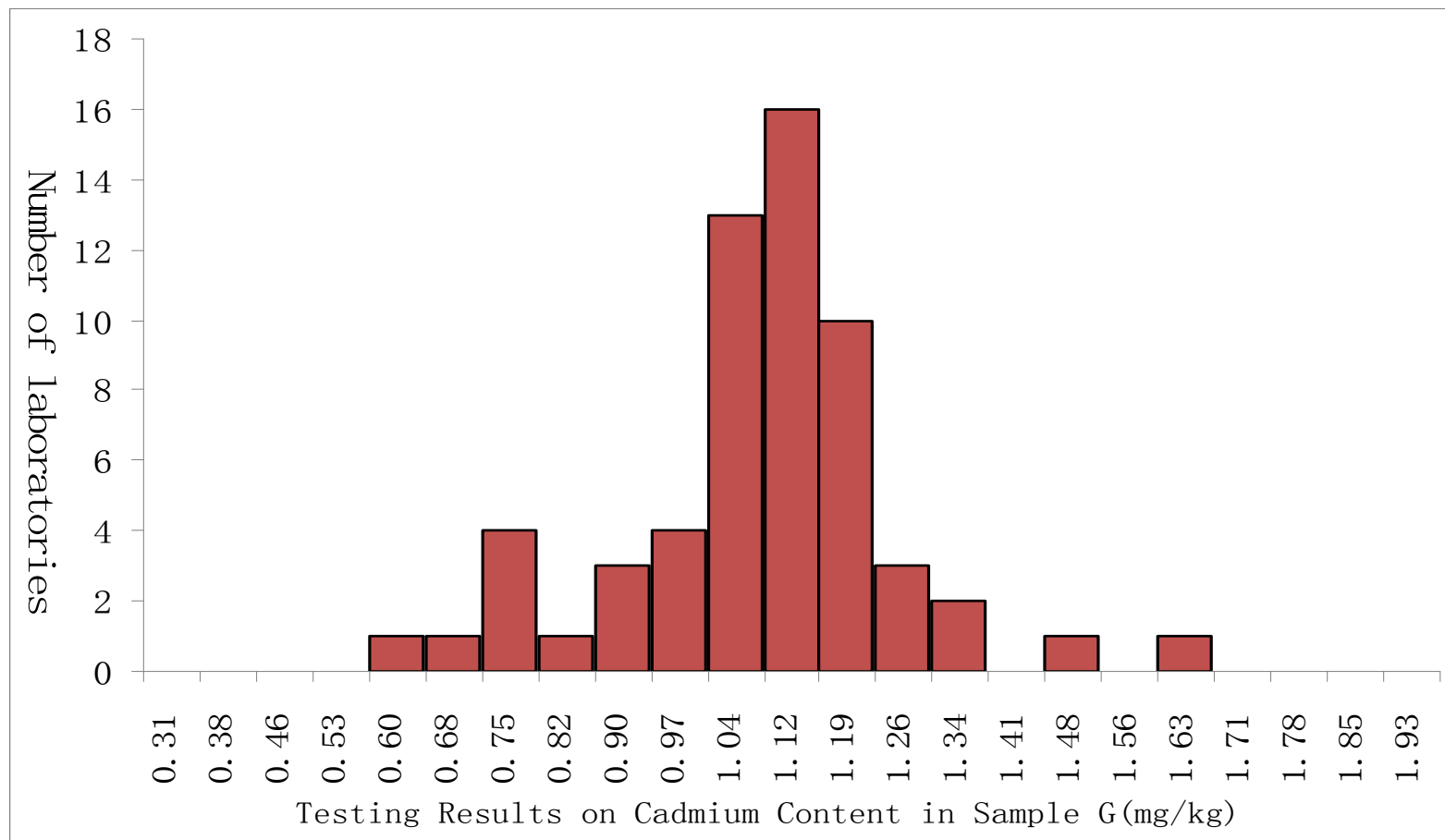
B.6 Distribution of Testing Results on Lead Content in Sample G



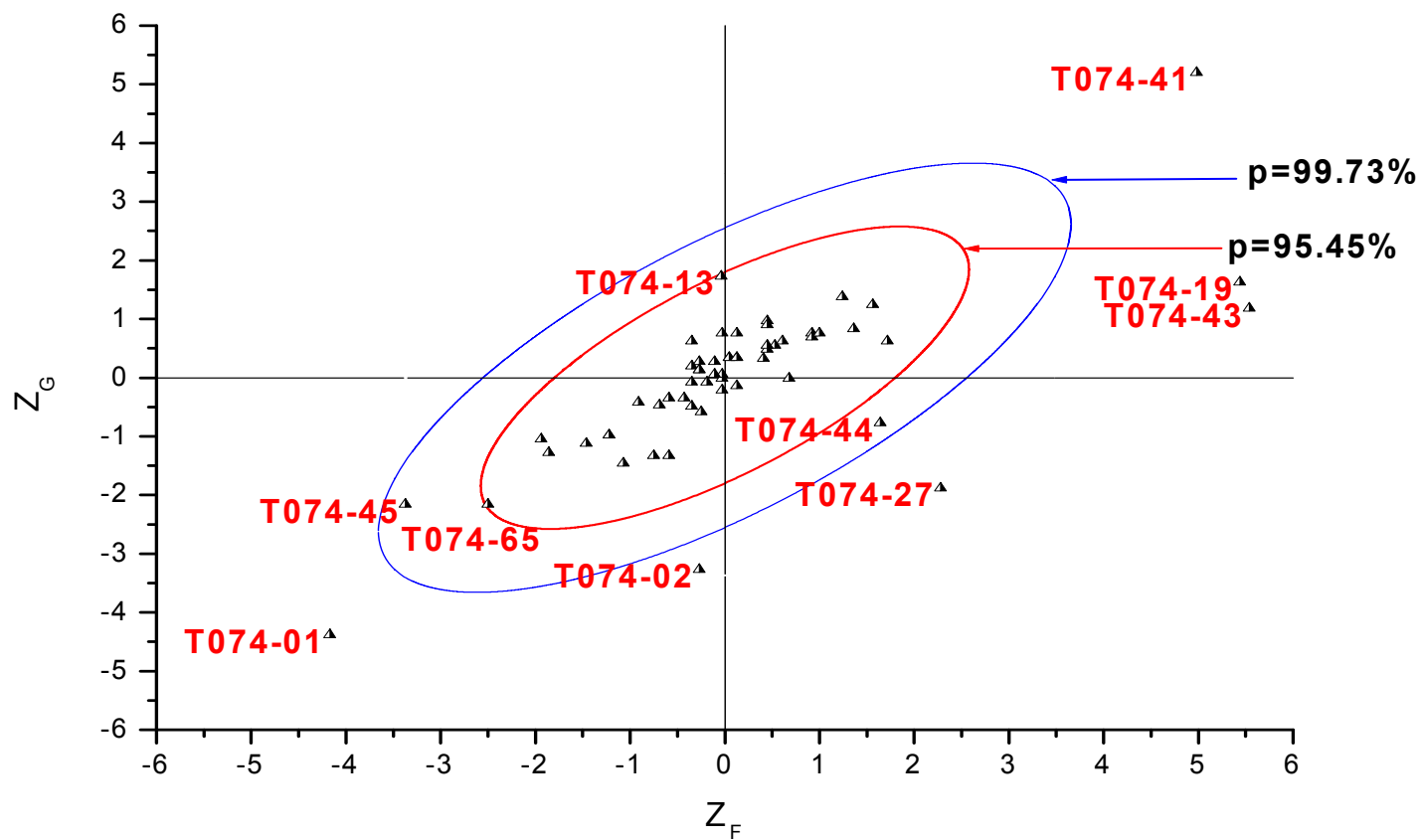
B.7 Distribution of Testing Results on Cadmium Content in Sample F



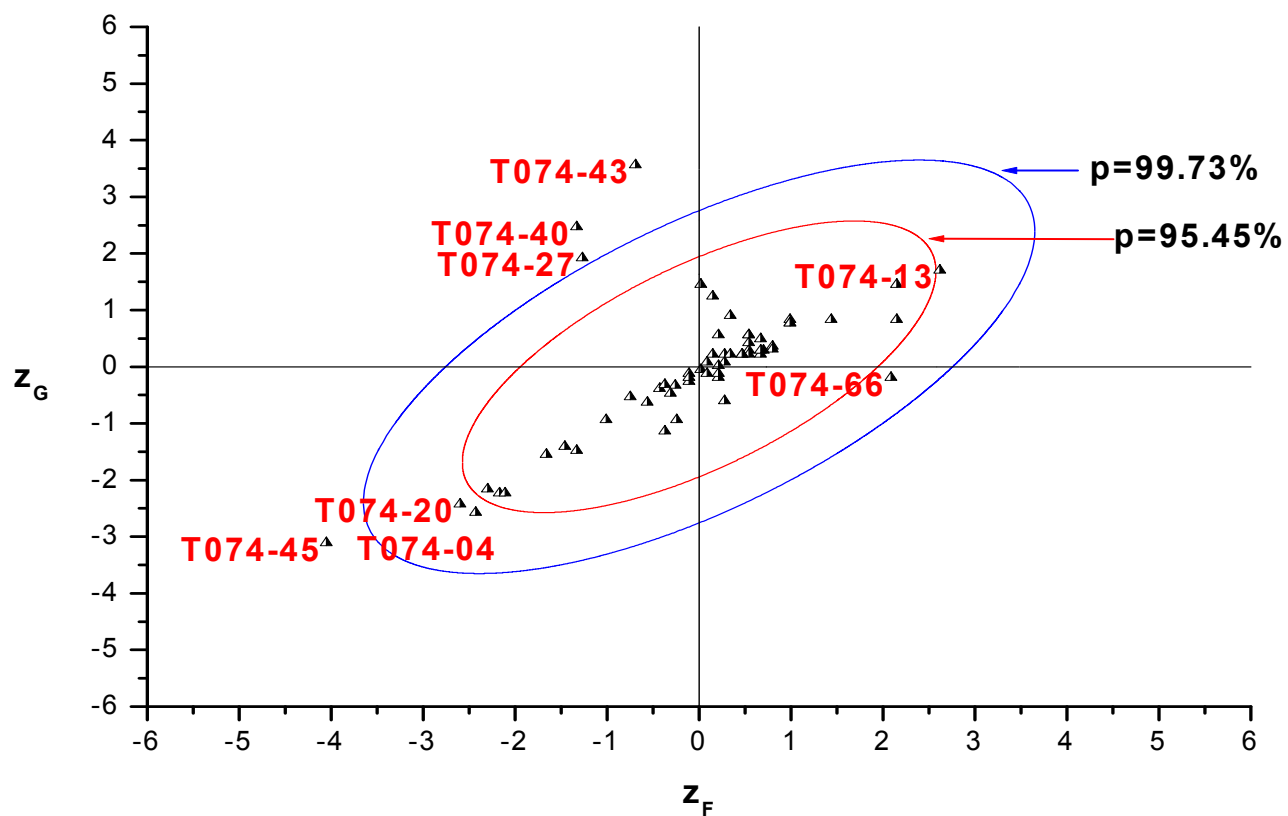
B.8 Distribution of Testing Results on Cadmium Content in Sample G



B.9 Youden Plot of z-scores of Lead



B.10 Youden Plot of z-scores of Cadmium



APPENDIX C

Homogeneity and Stability Checks

C.1 Homogeneity Testing

For each gross sample, 10 samples were randomly selected and tested. Each sample was divided into four test portions, all test portions were tested in random order on the repeatability condition, that is to say, the testing was carried out during a short period by one person using the same testing method and apparatus in one laboratory. The results on each sample are listed in Table C.1~C.4.

The sample average is $\bar{x}_i = \sum_{j=1}^n x_{ij} / n_i$

General average is $\bar{x} = \sum_{i=1}^m \bar{x}_i / m$

The between-samples sum of squares is $SS_1 = \sum_{i=1}^m n_i (\bar{x}_i - \bar{x})^2$

The between-samples mean square is $MS_1 = \frac{SS_1}{f_1}$

The within-sample sum of squares is $SS_2 = \sum_{i=1}^m \sum_{j=1}^{n_i} (x_{ij} - \bar{x}_i)^2$

The within-sample mean square is $MS_2 = \frac{SS_2}{f_2}$

Degrees of freedom is $f_1 = m - 1$

$$f_2 = N - m$$

The between-samples standard deviation is $S_s = \sqrt{(MS_1 - MS_2)/n}$

C.1 Homogeneity Testing Results of Lead in F Sample

Sample	x_{i1} (mg/kg)	x_{i2} (mg/kg)	x_{i3} (mg/kg)	x_{i4} (mg/kg)	$\bar{x}_i$ (mg/kg)
1	0.97	1.34	1.10	0.95	1.09
2	1.02	0.87	1.20	1.15	1.06
3	1.28	1.28	0.98	1.15	1.17
4	1.16	1.10	1.10	1.10	1.12
5	1.33	1.15	1.23	1.16	1.22
6	1.07	0.97	1.26	1.14	1.11
7	1.15	1.29	1.20	1.30	1.24
8	1.24	1.13	1.13	1.26	1.19
9	1.09	1.09	1.03	1.15	1.09
10	1.18	1.07	1.12	1.14	1.13
$\bar{\bar{x}} = 1.14$ mg/kg, $f_1 = 9$, $f_2 = 30$, $SS_1 = 0.127$, $MS_1 = 0.014$, $SS_2 = 0.334$, $MS_2 = 0.011$					
$S_s = 0.027$, Robust SD=0.13mg/kg					
S_s /Robust SD=0.21<0.30					

C.2 Homogeneity Testing Results of Lead in G Sample

Sample	x_{i1} (mg/kg)	x_{i2} (mg/kg)	x_{i3} (mg/kg)	x_{i4} (mg/kg)	$\bar{x}_i$ (mg/kg)
1	1.36	1.34	1.44	1.47	1.40
2	1.56	1.69	1.43	1.57	1.56
3	1.69	1.70	1.49	1.39	1.57
4	1.49	1.35	1.49	1.46	1.45
5	1.43	1.49	1.37	1.58	1.47
6	1.58	1.60	1.44	1.54	1.54
7	1.38	1.40	1.43	1.56	1.44
8	1.56	1.66	1.47	1.53	1.56
9	1.77	1.41	1.53	1.42	1.53
10	1.36	1.57	1.36	1.44	1.43
$\bar{\bar{x}} = 1.50$ mg/kg, $f_1 = 9$, $f_2 = 30$, $SS_1 = 0.140$, $MS_1 = 0.016$, $SS_2 = 0.278$, $MS_2 = 0.009$					
$S_s = 0.040$, Robust SD=0.14mg/kg,					
S_s /Robust SD=0.28<0.30					

C.3 Homogeneity Testing Results of Cadmium in F Sample

Sample	$x_{i1}(\text{mg/kg})$	$x_{i2}(\text{mg/kg})$	$x_{i3}(\text{mg/kg})$	$x_{i4}(\text{mg/kg})$	$\bar{x}_i (\text{mg/kg})$
1	1.50	1.61	1.58	1.60	1.57
2	1.58	1.63	1.74	1.78	1.68
3	1.68	1.68	1.67	1.68	1.68
4	1.72	1.63	1.71	1.59	1.66
5	1.74	1.68	1.59	1.55	1.64
6	1.71	1.63	1.68	1.69	1.68
7	1.55	1.78	1.75	1.51	1.65
8	1.76	1.70	1.63	1.65	1.69
9	1.52	1.59	1.61	1.62	1.59
10	1.57	1.68	1.62	1.61	1.62
$\bar{\bar{x}} = 1.64 \text{ mg/kg}, f_1 = 9 \quad f_2 = 30, SS_1 = 0.062, MS_1 = 0.007, SS_2 = 0.147, MS_2 = 0.005$					
$S_s = 0.023$, Robust SD=0.15mg/kg,					
$S_s / \text{Robust SD} = 0.15 < 0.30$					

C.4 Homogeneity Testing Results of Cadmium in G Sample

Sample	$x_{i1}(\text{mg/kg})$	$x_{i2}(\text{mg/kg})$	$x_{i3}(\text{mg/kg})$	$x_{i4}(\text{mg/kg})$	$\bar{x}_i (\text{mg/kg})$
1	1.31	1.35	1.35	1.36	1.34
2	1.17	1.16	1.31	1.31	1.24
3	1.35	1.35	1.36	1.28	1.34
4	1.30	1.27	1.41	1.37	1.34
5	1.33	1.21	1.20	1.36	1.28
6	1.27	1.35	1.34	1.30	1.32
7	1.22	1.37	1.31	1.28	1.30
8	1.22	1.29	1.27	1.23	1.25
9	1.12	1.19	1.28	1.30	1.22
10	1.40	1.42	1.31	1.23	1.34
$\bar{\bar{x}} = 1.28 \text{ mg/kg}, f_1 = 9 \quad f_2 = 30, SS_1 = 0.064, MS_1 = 0.007, SS_2 = 0.107, MS_2 = 0.004$					
$S_s = 0.030$, Robust SD=0.15mg/kg					
$S_s / \text{Robust SD} = 0.20 < 0.30$					

Comparing the between-samples standard deviation (s_s) with the corresponding standard deviation for proficiency assessment (Robust SD), all s_s are less than 0.3 Robust SD, it indicates that the variation between samples could be neglected in subsequently statistical analysis

and proficiency assessments, in another word, the samples are homogenous enough.

C.2 Stability Testing

For gross sample F and G, 12 packaged testing samples were selected for stability testing, respectively. The samples were placed at room temperature (18~25 degrees C) for 2 days, then putted into an attemperator with a temperature of 40 degrees C for 7 days and placed at about 4 degrees C until stability testing finished, on the date 15, 30 and 60 beginning from the day of homogeneity testing, every 3 samples per date were tested on Lead and Cadmium contents.

For all stability testing, the person, apparatus, testing method and laboratory were same as the homogeneity testing. The testing results of stability check are listed in Table C.5~C.8., every absolute differences from the general average of homogeneity tests were calculated using following formulae:

The sample average is $\bar{y}_t = (y_{t1} + y_{t2} + y_{t3} + y_{t4}) / 4$

General average is $\bar{\bar{y}} = \sum \bar{y}_t / g$

The difference between stability and homogeneity testing results is $|d| = |\bar{y} - \bar{\bar{x}}|$

Comparing with the standard deviations for proficiency assessment (Robust SD) in this program, all maximal absolute differences between the stability testing results and the corresponding homogeneity testing results ($|d|$) for each sample are less than 0.3 Robust SD, it indicates that the variation of samples could be neglected in subsequently statistical analysis and proficiency assessments.

As a conclusion, all samples are stable enough for the purposes of this program.

C.5 Stability Testing Results of Lead in F Sample

Day	y _{t1} (mg/kg)	y _{t2} (mg/kg)	y _{t3} (mg/kg)	y _{t4} (mg/kg)	$\bar{y}_t$ (mg/kg)	$\bar{\bar{y}}$ (mg/kg)	d
15	0.97	1.34	1.10	0.95	1.09	1.11	0.03
	1.02	0.87	1.20	1.15	0.95		
	1.28	1.28	0.98	1.15	1.28		
30	1.24	1.13	1.13	1.26	1.19	1.13	0.01
	1.09	1.09	1.03	1.15	1.09		
	1.18	1.07	1.12	1.14	1.13		
60	1.16	1.19	1.20	1.30	1.18	1.17	0.03
	1.25	1.13	1.13	1.23	1.19		
	1.19	1.09	1.23	1.15	1.14		
$\bar{\bar{x}}=1.14(\text{mg/kg})$, Robust SD =0.126, (mg/kg) max d / Robust SD =0.24<0.3							

C.6 Stability Testing Results of Lead in G Sample

Day	y _{t1} (mg/kg)	y _{t2} (mg/kg)	y _{t3} (mg/kg)	y _{t4} (mg/kg)	$\bar{y}_t$ (mg/kg)	$\bar{\bar{y}}$ (mg/kg)	d
15	1.36	1.34	1.44	1.47	1.40	1.52	0.02
	1.56	1.59	1.43	1.57	1.58		
	1.69	1.50	1.49	1.39	1.60		
30	1.38	1.41	1.32	1.55	1.40	1.52	0.02
	1.58	1.66	1.47	1.53	1.62		
	1.67	1.41	1.53	1.42	1.54		
60	1.38	1.40	1.43	1.56	1.39	1.51	0.01
	1.56	1.66	1.57	1.41	1.61		
	1.62	1.45	1.53	1.49	1.54		
$\bar{\bar{x}}=1.50(\text{mg/kg})$, Robust SD =0.144, (mg/kg) max d / Robust SD =0.14<0.3							

C.7 Stability Testing Results of Cadmium in F Sample

Day	y _{t1} (mg/kg)	y _{t2} (mg/kg)	y _{t3} (mg/kg)	y _{t4} (mg/kg)	$\bar{y}_t$ (mg/kg)	$\bar{\bar{y}}$ (mg/kg)	d
15	1.50	1.61	1.58	1.60	1.57	1.62	0.02
	1.58	1.63	1.74	1.78	1.61		
	1.68	1.68	1.67	1.68	1.68		
30	1.71	1.63	1.63	1.51	1.67	1.63	0.01
	1.55	1.68	1.61	1.65	1.62		
	1.58	1.62	1.54	1.62	1.60		
60	1.59	1.63	1.68	1.69	1.61	1.63	0.01
	1.55	1.58	1.65	1.51	1.57		
	1.71	1.70	1.63	1.65	1.71		
$\bar{\bar{x}}$ =1.64(mg/kg), Robust SD =0.155, (mg/kg) max d / Robust SD =0.13<0.3							

C.8 Stability Testing Results of Cadmium in G Sample

Day	y _{t1} (mg/kg)	y _{t2} (mg/kg)	y _{t3} (mg/kg)	y _{t4} (mg/kg)	$\bar{y}_t$ (mg/kg)	$\bar{\bar{y}}$ (mg/kg)	d
15	1.31	1.35	1.35	1.36	1.34	1.29	0.01
	1.17	1.16	1.31	1.31	1.17		
	1.35	1.35	1.36	1.28	1.35		
30	1.21	1.35	1.34	1.30	1.28	1.30	0.02
	1.28	1.37	1.31	1.28	1.33		
	1.32	1.29	1.27	1.33	1.31		
60	1.20	1.29	1.27	1.35	1.25	1.32	0.04
	1.32	1.29	1.28	1.30	1.31		
	1.40	1.42	1.31	1.23	1.41		
$\bar{\bar{x}}=1.28(\text{mg/kg})$, Robust SD =0.147, (mg/kg) $\max d /\text{Robust SD}=0.27<0.3$							

APPENDIX D

Instructions for Accreditation Bodies

1. Samples:

The Accreditation bodies (ABs) should receive two PT test items for each participating laboratory and instruction information (soft copy) from the PT coordinator. Upon receipt, ABs should carefully check the sample (number of bottles, physical conditions, etc.), and shall promptly acknowledge the LNCIQ by returning the “Receipt Form (for Accreditation Bodies)” electronically to syciqhlhlab@163.com. New sample(s) will be replaced for any missing and damaged bottles.

ABs are recommended to promptly distribute the test items to their nominated laboratories. The test items shall be stored at 0-4°C before dispatch.

ABs shall remind their nominated laboratories to carefully check the sample upon receipt and promptly acknowledge the LNCIQ by returning the “Receipt Form (for Participating Laboratories)” electronically to syciqhlhlab@163.com.

2. Results:

ABs shall inform their respective participating laboratories of completing Result Sheet and Analysis Information Form and submitting them electronically to the organizer on or before Nov.20 2011 to syciqhlhlab@163.com.

Notes: 1. Results submitted after the deadline will not be accepted by the organizers.

2. Due to possible poor transmission quality, results returned by fax are generally not acceptable).

3. Enquiries:

Please contact the coordinator for further enquiries at syciqhlhlab@163.com.

APPENDIX E

Instructions for Participants

Dear Participant

Your laboratory has been nominated by the laboratory accreditation body in your economy to participate in the Asia Pacific Laboratory Accreditation Cooperation (APLAC) Pb and Cd in Rice proficiency testing program T074. In this program, each laboratory has a unique code number. Your laboratory's code number is **T074-**_____. Considering the rules of protection of participants' confidentiality, the unique code number will represent your laboratory in the reports associated with this program.

Two samples have been sent to you in sealed bottles labeled as _____, _____. The weight of each sample is not less than 8g. Both concentrations of Pb and Cd in each sample are higher than 0.5mg/kg. Enclosed with the samples are the following documents (total 10 pages):

- Instructions to Participants
- Receipt Form
- Results Sheet
- Analysis Information Form

To ensure that results from this program can be analyzed properly, you are asked to follow carefully to the instructions below.

♪ Samples were sent by mail. You are requested to store them at 0-4°C immediately upon arrival. Once the sample bottles are opened, the tests must be performed immediately.

♪ You need to complete RECEIPT FORM **in English**, then **email** this completed form to the coordinator **on the same day** as you receive the samples.

♪ These samples are for laboratory use only, we will take no obligation or charge arising from other purposes.

♪ Sample Description: Two rice powder samples were provided to each participating laboratory. These two samples are at the different levels on Pb or Cd contents.



♪ Testing method: You should analyze Pb and/or Cd contents in the samples as you have applicated with any testing methods. However, the testing method you are going to use should be of the performance to accurately quantify 0.1mg/kg for Pb content and/or 0.05mg/kg for Cd content.

♪ Testing requested: The samples are for quantifying the contents of Pb and/or Cd in each sample. All samples must be analyzed and reported. It is recommended that each sample should be mixed, prior to test, thoroughly and respectively. It is suggested that minimum intake should be exceed 0.5g. Please record relevant information in TESTING RECORD FORMS in details, all you report will be benefit for analyzing testing results.

♪ Reporting results: For each sample, please report 4 test results and their mean result of Pb and/or Cd respectively on the RESULT SHEET. The test results should retain 2 decimal places.

Please fill out all relevant information, then email or fax the RESULT SHEET to the coordinator of this program and your accreditation body **NO LATER THAN THE DEADLINE 20th Nov. 2011.** And the TESTING RECORD FORMS and any supporting documents, e.g. measurement uncertainty report, should be submitted to your accreditation body before the deadline. At the same time, email or mail all the documents to:

Coordinator

Ms. Jin Yan

Address: No. 106, Dong Binhe Rd, Shenhe District, Shenyang, 110016,
P.R.China.

Tel: +86-24-24123659

Email: syciqhlhlab@163.com

PT Manager

Mr.Zhaipeijun

CNAS, The People's Republic of China



APPENDIX F

RECEIPT FORM

We are pleased to announce that in accordance with the distribution schedule of APLAC Pb and Cd in Rice proficiency testing program T074. We have dispatched the samples on Oct. 27, 2011 through TNT Express or FebEx, along with INSTRUCTIONS TO PARTICIPANTS, RECEIPT FORM, RESULT SHEET AND TESTING RECORD FORMS.

In order to monitor status of the samples, we kindly ask each participating laboratory on receipt of the samples to fill out this RECEIPT FORM and return a copy by **email URGENTLY to:**

Ms. Jin Yan

Tel: +86-24-24123659

E-mail: syciqlhlab@163.com

Thank you in advance for your cooperation.

NAME OF LABORATORY:

LAB CODE: T074-_____

DATE SAMPLE RECEIVED: _____ **SAMPLE AMOUNT:** _____

The samples are labelled as follows: _____, _____

Inspect the packaging. Was there any visible damage? ☐ Yes ☐ No

If yes, or any other problem is available, describe the problem below.

Signature:

Date:

APPENDIX G

Results Sheet

Lab Code: T074—

SAMPLE RECEIVED DATE: _____	REPORT DATE: _____
SAMPLE PROCESSED DATE: From _____ to _____	

Results of Lead Contents in Samples

Sample No.	Testing Results(mg/kg)				Mean Result with Uncertainty (mg/kg)	Limit of Quatitation (mg/kg)
	1	2	3	4		

Results of Cadmium Contents in Samples

Sample No.	Testing Results(mg/kg)				Mean Result with Uncertainty (mg/kg)	Limit of Quatitation (mg/kg)
	1	2	3	4		

Signature:

Date:

Return this Results Sheet (including the Testing Record Form) by **no later than the deadline Nov. 20, 2011** (we will confirm it by local postmark) by email, and then mail to:

Ms. Jin Yan

Adress: No. 106, Dong Binhe Rd, Shenhe District, Shenyang, 110016, P.R.China.

Tel: +86-24-24123659

Email: syciqlhlab@163.com

APPENDIX H

Analysis Information Form

Lab Code: T 074—

Is the method used for the results submitted in your scope of accreditation?		lead		☐ yes	☐ no
		cadmium		☐ yes	☐ no
Reference:	lead	☐ standard/ ☐ journal/ ☐ book	year	No./Vol /Edition	pages
	cadmium	☐ standard/ ☐ journal/ ☐ book	year	No./Vol /Edition	pages
Protocol for Quantification	lead	☐ External Standards ☐ Internal Standards ☐ Others (specify): _____			
	cadmium	☐ External Standards ☐ Internal Standards ☐ Others (specify): _____			
Reference Materials	lead	Supplier(s): Purity:			
	cadmium	Supplier(s): Purity:			
Reagents and Materials	lead				
	cadmium				
Sample Treatment	lead	Method:			

		Instruments used:
	cadmium	Method: Instruments used:
Operating Condition	lead	Equipment Model: _____ Detector: _____ Wavelength: _____ Hollow Cathode Lamp Current: _____ Eliminating Background Mode: _____ Atomization Temperature: _____ °C Injection Volume: _____ μL
	cadmium	Equipment Model: _____ Detector: _____ Wavelength: _____ Hollow Cathode Lamp Current: _____ Eliminating Background Mode: _____ Atomization Temperature: _____ °C Injection Volume: _____ μL
Testing Sample	lead	Mass of Test Portions (in g) _____
	cadmium	Mass of Test Portions (in g) _____
Spectrogram		Spectrograms Attached: _____ Pages
Calculating Formulae		lead cadmium

Signature:
Date: