HERBAL MEDICINE PROFICIENCY TESTING PROGRAMME

(APLAC – T043)

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1. Introduction

1.1. This report summarises the results of the proficiency testing programme on the analysis for the levels of Cadmium and Lead in herbal medicine.

1.2. The programme was organised by the Government Laboratory of the HKSAR (GL), in collaboration with the Hong Kong Accreditation Service (HKAS), under the auspices of the Asia-Pacific Laboratory Accreditation Co-operation (APLAC).

1.3. A total of 38 laboratories participated in this programme and are listed in Appendix I. Each laboratory is identified in this report by a unique code.

2. Objectives

2.1. The programme aims at providing a means for participating laboratories to evaluate their performance in the analysis for the levels of cadmium and lead in herbal medicine.

2.2. Test results from all participating laboratories are evaluated according to the robust statistical techniques outlined in ISO/DIS 13528 "Statistical Methods for use in Proficiency Testing by Interlaboratory"[9.1] and Analytical Methods Committee of Royal Society of Chemistry[9.2]. Z-score is provided as a numerical indicator to reveal each participating laboratory's proficiency relative to other laboratories.

3. Samples

3.1. About 10 kg of Guangjinqiancao (廣金錢草) [*Herba Desmodii Styracifolii*] were purchased from the local market and authenticated by the Government Laboratory of the HKSAR. After being washed thoroughly to remove foreign matters and soil, the herb was freeze-dried. The dried herb was powdered, sieved through a 100μm sieve and mixed thoroughly. About 25 g of the powder was dispensed into each of 100 plastic bottles, which was sealed under vacuum and marked with a randomly-assigned unique serial number.

3.2. The homogeneity and stability of the test samples were determined in accordance with the procedures stipulated in Appendix II and the results were found satisfactory.

3.2 50 sets of powdered samples together with "Instructions to accreditation bodies", "*Receipt form*", "*Instructions to participating laboratories*" and "*Results proforma*" were sent to HKAS in April 2005 for redirection to participating accreditation bodies. A specimen copy of these instructions is given in Appendix II. Each participating laboratory was provided with one bottle of test sample by its accreditation body.

3.3 38 participating laboratories returned their analysis results.

4. Test procedures

4.1 Each participating laboratory was required to determine the levels of Cadmium and Lead in the powder according to the suggested method given in the "*Instructions to participating laboratories*" or other appropriate validated methods.

4.2 A summary of the techniques employed by the individual participating laboratories is presented in Table 3. The major analytical techniques used by the participating laboratories are Inductively coupled plasma - mass spectrometry (ICP-MS), Inductively coupled plasma - optical emission spectroscopy (ICP-OES), atomic absorption spectroscopy (AAS), etc. For the sample preparation, most of the laboratories employed microwave-assisted digestion using nitric acid or nitric acid/hydrogen peroxide/hydrochloric acid.

5. Test results and statistical treatment

5.1. Test results

5.1.1. Test results submitted by the participating laboratories are summarised in Tables 1 & 2 in Appendix IV.

5.1.2. Calculated means and standard deviations are given in Tables 4 & 5 respectively in Appendix IV. The histograms of the data are given in figures 1 to 4.

5.1.3 The mean of laboratories' means (m) and standard deviation of the laboratories' means (s) computed by robust statistics are given in Table 6 in Appendix V.

5.3. Performance evaluation

5.3.1. The performance of each participating laboratory was evaluated by means of a performance index, Z-score which is calculated as:

$$Z = \frac{x_i - m}{s}$$

where

x_i = laboratory test results
m = computed mean
s = computed standard deviation

5.3.2 The Z-scores are tabulated in Table 7 in Appendix V and illustrated graphically in Figures 1 & 2.

6. Conclusion

The performance of the majority of participating laboratories was satisfactory. More than 90% of the test results reported by the participating laboratories achieved a satisfactory Z-score.

7. Recommendations

7.1 The Z-score is commonly interpreted as follows:

(a) $|Z| \leq 2$ Satisfactory

(b) $2 < |Z| < 3$ Questionable

(c) $|Z| \geq 3$ Unsatisfactory

7.2 Z-scores marked with asterisk (*) are considered unsatisfactory (i.e. $|Z| \geq 3$) and the laboratories concerned should investigate their results.

7.3 Laboratories having a Z-score in the range $2 < |Z| < 3$ are encouraged to review their results.

8. Future programmes

8.1. Interlaboratory proficiency testing programmes allow laboratories to demonstrate the comparability of their data. The Government Laboratory of Hong Kong SAR is prepared to conduct similar proficiency testing programmes to assist laboratories in achieving this objective.

8.2. Suggestions and comments relating to this proficiency testing programme are welcome.

9. References

9.1. ISO/DIS 13528:2005, "Statistical Methods for use in Proficiency Testing by Interlaboratory", 2005, Geneva.

9.2. Analytical Methods Committee of RSC, *Analyst*, Vol.114, 1989, 1693-1702.International Harmonized Protocol for Proficiency

9.3. Testing of (Chemical) Analytical Laboratories, *JAOAC*, Vol.76, No.4, 1993, 926-940.

9.4. R.E. Lawn, M. Thompson, R.F. Walker, Proficiency Testing in Analytical Chemistry, Royal Society of Chemistry, 1997, UK.

9.5. J.N. Miller, J.C. Miller, Statistics and Chemometrics for Analytical Chemistry, 4th edition, 2000, Prentice Hill.

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Assessing homogeneity and stability of the test materials

A. Homogeneity test

1. Ten bottles of powder were randomly selected from a total of 100 bottles.
2. The powder in each selected bottle was thoroughly mixed and three test portions (sub-samples) were drawn from each bottle.
3. The 30 sub-samples were analyzed for the levels of cadmium and lead in a random order under same conditions.
4. The sampling variance (S_s^2) and analytical variance (S_a^2) were estimated based on one-way analysis of variance (ANOVA), without exclusion of outliers, using the following equations:

$$\bar{x}_i = \frac{1}{n}\sum_{j=1}^{n} x_{ij}$$

$$\bar{\bar{x}} = \frac{1}{k}\sum_{i=1}^{k} \bar{x}_i$$

$$SS_1 = n \cdot \sum_{i=1}^{k} (\bar{x}_i - \bar{\bar{x}})^2$$

$$SS_2 = \sum_{i=1}^{k}\sum_{j=1}^{n} (x_{ij} - \bar{x}_i)^2$$

sampling variance (S_s^2) = SS_1 / (k-1)

analytical variance (S_a^2) = SS_2 / k(n-1)

where:

k = number of samples

n = number of replicates per sample

5. The values of $\bar{\bar{x}}$, S_s, S_a, k and the results of F-test are given below:

Element	$\bar{\bar{x}}$ **, mg/kg**	S_s^2	S_a^2	**F**	$F_{critical}$
Cadmium	0.296	0.000023	0.000017	1.319	2.393
Lead	1.41	0.001437	0.002077	0.692	2.393

6. Since the test statistic F is less than the critical value, there was no significant difference between the sampling and analytical variances, i.e. the sample could be considered homogeneous.

B. Stability tests

1. The stability of the samples was monitored regularly over an extended period (from January to November 2005) covering the time when the samples were analyzed by the participants.

2. Samples selected for the stability test were stored under conditions as given in the "instructions to the participants".

4. Three samples were randomly selected in each monitoring exercise and analyzed in duplicate.

5. The target value for standard deviation of the proficiency test (σ_P) was taken to be 0.1 (i.e 10% of the mean value).

6. The summary of the results is given below:

Element	$\overline{x1}$ **(Jan)**	$\overline{x2}$ **(Jun)**	$\overline{x3}$ **(Nov)**	$\lvert\overline{x1}-\overline{x2}\rvert$	$\lvert\overline{x1}-\overline{x3}\rvert$	σ_P	**0.3**σ_P
Cadmium	0.289	0.292	0.293	0.003	0.004	0.030	0.009
Lead	1.480	1.455	1.468	0.025	0.012	0.148	0.044

7. Since the differences between the results obtained over ten months were found to be less than 0.3 of the target value for standard deviation of the proficiency test (σ_P), the stability of the samples was considered satisfactory.

HONG KONG GOVERNMENT LABORATORY

IN COLLABORATION WITH

HONG KONG ACCREDITATION SERVICE

APLAC T043 – HERBAL MEDICINE PROFICIENCY TESTING PROGRAM

INSTRUCTIONS TO ACCREDITATION BODIES

1. Samples

Each participating laboratory is given a sample bottle containing about 20 g of sample powder.

Upon receipt of the sample bottle from HKAS, participating accreditation bodies should check the sample for damage, if any, and acknowledge receipt of the sample by returning the Receipt Form (FORM 02) to HKAS via e-mail (eyslee@itc.gov.hk), fax (+852 2824 1302).

2. Tests to be performed

Each participating laboratory is required to determine the levels of Cadmium and Lead (in mg/kg) in the sample powder on a dried weight basis. The determination should be conducted in triplicate. The drying testing shall be carried out on a separate sample portion by drying at 105℃ for at least three hours.

3. Testing method

Suggested Method: Samples are digested either by low- or high-pressure microwave assisted nitric acid digestion. After dilution with water, the metals are determined using inductively coupled plasma-mass spectrometry (ICP-MS).
Requirement of the method used by the participant: The digestion techniques used shall be strong enough to dissolve almost all elements of concern from many types of Chinese medicine samples.

4. Sample distribution

The samples, Instructions to Laboratories (FORM 03) and Results Proforma (FORM 04) should be distributed to participating laboratories as soon as possible.

Laboratories will have about four weeks to analyse the samples.

5. Documents to be submitted

Accreditation bodies should collect the Result Proforma (FORM 05) from the participating laboratories on or before 31 July 2005, and forward the Results Proforma to HOKLAS before 15 August 2005.

6. Enquiries

Please contact:- Miss Linda Lam
HKAS
36/F, Immigration Tower, 7 Gloucester Road, Hong Kong
Fax: +852 2824 1302
Tel.: +852 2829 4808
Email: hkas@itc.gov.hk

HONG KONG GOVERNMENT LABORATORY

IN COLLABORATION WITH

HONG KONG ACCREDITATION SERVICE

APLAC T043 – HERBAL MEDICINE PROFICIENCY TESTING PROGRAM

RECEIPT FORM

<To be completed by Participating Accreditation Body>

From: _____
Participating Accreditation Body

_____
Contact Person

Fax: _____

Tel.: _____

Email: _____

Date: _____

To: HKAS,
36/F Immigration Tower,
7 Gloucester Road,
Hong Kong.

Attn.: Mr. Eric Y S Lee

Fax: +852 2824 1302

Tel.: +852 2829 4804

Email: eyslee@itc.gov.hk

Please complete the shaded area.

This is to acknowledge the receipt of the APLAC sample.

After inspection, the sample was found (☐ damaged / ☐ intact)* and were deemed (☐ suitable / ☐ not suitable)* for testing.

REMARK(s) : _____

* Click as appropriate

HONG KONG GOVERNMENT LABORATORY

IN COLLABORATION WITH

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APLAC T043 – HERBAL MEDICINE PROFICIENCY TESTING PROGRAM

INSTRUCTIONS TO PARTICIPATING LABORATORIES

Laboratory Sample Number: .....
Laboratory Code: .....

1. Samples

Please check whether you are provided with a sample bottle containing about 20 g of sample powder.

2. Tests to be performed

Each participating laboratory is required to determine the levels of Cadmium and Lead (in mg/kg) in the sample powder on a dried weight basis. The determination should be conducted in triplicate. The drying testing shall be carried out on a separate sample portion by drying at 105℃ for at least three hours.

3. Testing method

Suggested Method: Samples are digested either by low- or high-pressure microwave assisted nitric acid digestion. After dilution with water, the metals are determined using inductively coupled plasma-mass spectrometry (ICP-MS).
Requirement of the method used by the participant: The digestion techniques used shall be strong enough to dissolve almost all elements of concern from many types of Chinese medicine samples.

4. Reporting

Report all the replicate data in the Results Proforma (FORM 04) and complete both Part I & II.

5. Documents to be submitted

Submit the completed Results Proforma to your accreditation body on or before 31July 2005. via e-mail, fax (+852 2824 1302)

6. Enquires

Please contact your accreditation body for general enquiry concerning the program. Detailed information of the program may be obtained from:-

Miss Linda Lam (HKAS)

36/F, Immigration Tower, 7 Gloucester Road, Hong Kong
Fax: +852 2824 1302
Tel.: +852 2829 4808
Email: hkas@itc.gov.hk

HONG KONG GOVERNMENT LABORATORY

IN COLLABORATION WITH

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APLAC T043 – HERBAL MEDICINE PROFICIENCY TESTING PROGRAM

RESULTS PROFORMA

<Both Part I & II should be completed>

Laboratory Sample Number: .....

Laboratory Code: ..... Date of Analysis: .....

Name of Laboratory: .....

Part I: Analytical Data

Cadmium and Lead in sample powder

(a) Analytical results:

Ingredients	Level, mg/kg (**based on dried weight**)[§]		
	Replicate 1	Replicate 2	Replicate 3
Cadmium	.....	.....	.....
Lead	.....	.....	.....

(b) Uncertainty:

Ingredients	Mean[§], mg/kg	Expanded Uncertainty, *U*, mg/kg	Coverage Factor, *k*
Cadmium	.....	.....	.....
Lead	.....	.....	.....

[§] Report values to 3 significant Figure or 3 decimal places, whichever is appropriate

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Part II: Test Parameters

☐ Click as appropriate

1. Analytical technique and instrument model (s):
   - ☐ ICP-MS (model .....)
   - ☐ AAS (model .....)
   - ☐ ICP-OES (model .....)
   - ☐ Others (with model) ..... *(Please specify)*

2. Digestion method:
   - ☐ Microwave-assisted digestion (make and model of the microwave .....)
   - ☐ Others ..... *(Please specify)*

3. Digestion medium: ..... *(Please specify)*

4. Digestion programme/ condition: .....

5. Other comments: .....

Note: The accreditation body under which your laboratory is accredited may be informed of your results and performance in this programme.

Submission of this Results Proforma via:

☐ E-mail ☐ Fax

Completed by: ...../..... Date: .....

(Name with signature / Post

Fax To : Mr. Eric Lee
Fax No. : 852 2824 1302
Email : eyslee@itc.gov.hk

APLAC T043 – HERBAL MEDICINE PROFICIENCY TESTING PROGRAMME
Nominated Participating Laboratories

Contact *Lab Name* *Physical Address* *Phone* *Fax* *Accredited for tests YES/NO*	***Contact*** *Lab Name* *Physical Address* *Phone* *Fax* *Accredited for tests YES/NO*
Contact *Lab Name* *Physical Address* *Phone* *Fax* *Accredited for tests YES/NO*	***Contact*** *Lab Name* *Physical Address* *Phone* *Fax* *Accredited for tests YES/NO*

From : *(NATIONAL LABORATORY ACCREDITATION BODY)*

Name

Organisation

Postal Address

Phone

Email

Table 1 Original data – Cadmium

Lab Code	Sample Code	Analytical Results Cadmium (mg/kg)				Uncertainty Cadmium (mg/kg)	
		Replicate 1	Replicate 2	Replicate 3	Mean	Expanded Uncertainty	Coverage Factor
1	GL1001	0.257	0.251	0.197	0.235	0.12	2
2	GL1002	0.250	0.246	0.246	0.247	0.068	2
3	GL1003	0.297	0.298	0.307	0.301	0.069	2
4	GL1004	<0.400	<0.400	<0.400	<0.400	--	2
5	GL1005	0.243	0.243	0.237	0.241	0.039	2
6	GL1007	0.290	0.290	0.280	0.287	0.0252	2
7	GL1008	0.276	0.271	0.275	0.274	0.038	2
8	GL1011A	0.291	0.281	0.281	0.284	0.028	2
9	GL1011B	0.306	0.295	0.328	0.310	0.034	2
10	GL1011C	0.269	0.261	0.251	0.260	0.037	2
11	GL1012	0.231	0.271	0.251	0.251	0.02	2
12	GL1013	0.189	0.196	0.191	0.192	0.015	4.3
13	GL 1019	0.2837	0.3283	0.2792	0.297	0.039	2
14	GL 1021	0.159	0.158	0.159	0.159	0.002	2
15	GL 1020	0.310	0.327	0.342	0.326	0.049	2
16	GL 1018	0.165	0.148	0.162	0.158	0.031	2
17	GL1016	0.283	0.236	0.258	0.259	--	--
18	463970	0.278	0.284	0.277	0.280	0.028	--
19	370787/1	0.325	0.299	0.313	0.312	0.031	2
20	GL1023	--	--	--	--	--	--
21	GL1024	0.373	0.362	0.378	0.371	0.016	2
22	GL1025	0.184	0.188	0.186	0.186	0.004	2
23	GL1029	0.331	0.335	0.336	0.334	0.0293	2
24	GL1030	0.253	0.257	0.25	0.253	0.009	2
25	GL1031	0.254	0.321	0.306	0.294	0.042	2
26	GL1032	0.342	0.364	0.32	0.342	0.039	2
27	GL1034	0.271	0.263	0.265	0.266	0.015	2
28	GL1036	0.28	0.23	0.19	0.233	0.05	2
29	GL1037	0.262	0.253	0.304	0.273	0.05	2
30	GL1039	0.214	0.238	0.221	0.224	0.056	2
31	GL1040	0.245	0.225	0.223	0.231	0.09	2
32	GL1041	0.21	0.211	0.211	0.211	0.026	2
33	GL1042	0.349	0.311	0.335	0.332	0.073	2
34	GL1043	0.292	0.295	0.296	0.294	0.021	2
35	GL1044	--	--	--	--	--	--
36	GL1046	0.283	0.333	0.352	0.323	--	--
37	GL1048	0.271	0.275	0.276	0.274	0.039	2
38	GL1049	0.262	0.263	0.298	0.274	0.0658	2

--- denotes that the laboratory did not submit any data.

Table 2 Original data – Lead

Lab Code	Sample Code	Analytical Results Lead (mg/kg)				Uncertainty Lead (mg/kg)	
		Replicate 1	Replicate 2	Replicate 3	Mean	Expanded Uncertainty	Coverage Factor
1	GL1001	1.32	1.44	1.09	1.283	0.095	2
2	GL1002	1.39	1.38	1.400	1.390	0.367	2
3	GL1003	1.47	1.47	1.45	1.463	0.350	2
4	GL1004	1.47	1.46	1.48	1.470	0.382	2
5	GL1005	1.549	1.539	1.571	1.553	0.216	2
6	GL1007	1.42	1.41	1.39	1.407	0.147	2
7	GL1008	1.44	1.48	1.46	1.460	0.201	
8	GL1011A	--	--	--	--	--	--
9	GL1011B	--	--	--	--	--	--
10	GL1011C	1.55	1.29	1.44	1.427	0.276	2
11	GL1012	1.415	1.386	1.444	1.415	0.03	2
12	GL1013	--	--	--	--	--	--
13	GL 1019	3.345	3.393	3.337	3.358	0.045	2
14	GL 1021	1.508	1.503	1.511	1.507	0.005	2
15	GL 1020	1.396	1.403	1.341	1.380	0.210	2
16	GL 1018	1.576	1.350	1.587	1.504	0.033	2
17	GL1016	1.40	1.22	1.34	1.320	--	--
18	463970	1.39	1.40	1.41	1.400	0.14	--
19	370787/1	1.32	1.34	1.36	1.340	0.13	2
20	GL1023	5.98	5.94	5.30	5.740	0.4	2
21	GL1024	1.094	1.167	1.175	1.145	0.089	2
22	GL1025	1.107	1.134	1.13	1.124	0.032	2
23	GL1029	1.45	1.44	1.37	1.420	0.133	2
24	GL1030	1.476	1.468	1.364	1.436	0.088	2
25	GL1031	1.3	1.56	1.24	1.367	0.23	2
26	GL1032	1.4	1.51	1.58	1.497	0.17	2
27	GL1034	1.65	1.58	1.63	1.620	0.081	2
28	GL1036	1.8	1.3	1.4	1.500	0.3	2
29	GL1037	1.82	1.9	1.91	1.877	0.2	2
30	GL1039	1.008	0.985	0.866	0.953	0.305	2
31	GL1040	1.11	0.837	0.892	0.946	0.31	2
32	GL1041	1.32	1.22	1.22	1.253	0.126	2
33	GL1042	1.545	1.415	1.41	1.457	0.35	2
34	GL1043	1.432	1.462	1.414	1.436	0.62	2
35	GL1044	1.4	1.31	1.33	1.347	0.27	2
36	GL1046	1.62	1.6	1.52	1.580	--	--
37	GL1048	1.54	1.54	1.54	1.540	0.07	2
38	GL1049	1.412	1.381	1.254	1.349	0.4182	2

-- denotes that the laboratory did not submit any data.

Table 3 - Techniques used by individual laboratories

Lab Code	Analytical technique	Digestion method	Digestion medium
1	ICP-OES	Microwave-assisted digestion	Nitric acid and hydrogen peroxide
2	ICP-MS	Microwave-assisted digestion	Nitric acid and hydrogen peroxide
3	ICP-MS	Microwave-assisted digestion	Nitric acid and hydrochloric acid
4	ICP-MS	Microwave-assisted digestion	Nitric acid
5	ICP-MS	Microwave-assisted digestion	Nitric acid
6	ICP-MS and AAS	Microwave-assisted digestion	Nitric acid
7	ICP-MS	Microwave-assisted digestion	Nitric acid
8	AAS(SSGF)	No digestion	N.A.
9	RNAA	Open system on electrical plate	Sulphuric acid and hydrogen peroxide
10	ICP-OES	Pressure digestor	Nitric acid
11	ICP-MS	Microwave-assisted digestion	Nitric acid and hydrogen peroxide
12	ICP-OES	Microwave-assisted digestion	Nitric acid, hydrogen peroxide and hydrofluoric acid
13	AAS	Dry ashing	N.A.
14	AAS	Muffle furnace	Nitric acid and Sulphuric acid
15	AAS	Microwave-assisted digestion	Nitric acid and hydrogen peroxide
16	AAS	Muffle furnace	Nitric acid
17	ICP-MS	Microwave-assisted digestion	Nitric acid
18	ICP-MS	Water bath	Nitric acid
19	ICP-MS	Hot block	Nitric acid and hydrochloric acid
20	AAS	AOAC wet ashing	Nitric acid and Perchloric acid
21	AAS	Dry ashed	N.A.
22	ICP-MS	Microwave-assisted digestion	Nitric acid
23	AAS	Microwave-assisted digestion	Nitric acid and hydrogen peroxide
24	AAS	Microwave-assisted digestion	Nitric acid
25	AAS	Wet ashing	Nitric acid
26	AAS	High-pressure digestion	Nitric acid
27	AAS	Graphite block	Nitric acid and hydrochloric acid
28	AAS	Microwave-assisted digestion	Nitric acid
29	AAS	Microwave-assisted digestion	Nitric acid
30	AAS	Dry ashing in muffle furnace	N.A.
31	ICP-MS	Microwave-assisted digestion	Nitric acid and hydrogen peroxide
32	ICP-OES	Microwave-assisted digestion	Nitric acid and hydrogen peroxide
33	ICP-MS	Microwave-assisted digestion	Nitric acid and hydrogen peroxide
34	AAS	Microwave-assisted digestion	Nitric acid
35	ICP-MS	Microwave-assisted digestion	Nitric acid
36	AAS	Microwave-assisted digestion	Nitric acid and hydrogen peroxide
37	ICP-MS	High pressure asher	Nitric acid
38	ICP-MS	Microwave-assisted digestion	Nitric acid and hydrogen peroxide

Notes:
1. The above summary is based on the information given in Part II of the *Results Proforma*
2. AAS(SSGF): Solid sample graphite furnace atomic absorption spectrometry
3. RNAA: Radiochemical Neutron Activation Analysis.
4. N.A.: Not applicable.

Table 4 Mean values

	Cadmium	**Lead**
Lab Code	Mean Value (mg/kg)	Mean Value (mg/kg)
1	0.235	1.28
2	0.247	1.39
3	0.301	1.46
4	<0.400	1.47
5	0.241	1.55
6	0.287	1.41
7	0.274	1.46
8	0.284	--
9	0.310	--
10	0.260	1.43
11	0.251	1.42
12	0.192	--
13	0.297	3.36
14	0.159	1.51
15	0.326	1.38
16	0.158	1.50
17	0.259	1.32
18	0.280	1.40
19	0.312	1.34
20	--	5.74
21	0.371	1.14
22	0.186	1.12
23	0.334	1.42
24	0.253	1.44
25	0.294	1.37
26	0.342	1.50
27	0.266	1.62
28	0.233	1.50
29	0.273	1.88
30	0.224	0.953
31	0.231	0.946
32	0.211	1.25
33	0.332	1.46
34	0.294	1.44
35	--	1.35
36	0.323	1.58
37	0.274	1.54
38	0.274	1.35

-- denotes that the laboratory did not submit any data

Table 5 Standard deviations

	Cadmium	**Lead**
Lab Code	Standard deviation (mg/kg)	Standard deviation (mg/kg)
1	0.033	0.178
2	0.002	0.010
3	0.006	0.012
4	--	0.010
5	0.003	0.016
6	0.006	0.015
7	0.003	0.020
8	0.006	--
9	0.017	--
10	0.009	0.131
11	0.020	0.029
12	0.004	--
13	0.027	0.030
14	0.001	0.004
15	0.016	0.034
16	0.009	0.134
17	0.024	0.092
18	0.004	0.010
19	0.013	0.020
20	--	0.382
21	0.008	0.045
22	0.002	0.015
23	0.003	0.044
24	0.004	0.062
25	0.035	0.170
26	0.022	0.091
27	0.004	0.036
28	0.045	0.265
29	0.027	0.049
30	0.012	0.076
31	0.012	0.144
32	0.001	0.058
33	0.019	0.077
34	0.002	0.024
35	--	0.047
36	0.036	0.053
37	0.003	0.000
38	0.021	0.084

-- denotes that the laboratory did not submit any data

Table 6 Computed values of mean of laboratories' means and standard deviation of laboratories' means

All test results were included in the robust statistical treatment[9.1, 9.2]. The statistical parameters are reported as follows:

	Cadmium	Lead
Mean of laboratories' means (m), mg/kg	0.270	1.43
Standard deviation of laboratories means (s), mg/kg	0.0492	0.136
Relative standard deviation of laboratories' means (rsd), %	18.2	9.5

Table 7 Z-scores

	Cadmium	Lead
Lab Code	Z-Score	Z-Score
1	-0.71	-1.0
2	-0.46	-0.26
3	0.62	0.28
4	--	0.32
5	-0.59	0.93
6	0.34	-0.14
7	0.08	0.25
8	0.29	--
9	0.80	--
10	-0.20	0.01
11	-0.39	-0.08
12	-1.6	--
13	0.55	14 *
14	-2.3	0.60
15	1.1	-0.33
16	-2.3	0.58
17	-0.23	-0.77
18	0.19	-0.19
19	0.86	-0.63
20	--	31*
21	2.1	-2.0
22	-1.7	-2.2
23	1.3	-0.04
24	-0.34	0.08
25	0.48	-0.43
26	1.5	0.52
27	-0.08	1.4
28	-0.75	0.54
29	0.06	3.3 *
30	-0.93	-3.5 *
31	-0.80	-3.5 *
32	-1.2	-1.3
33	1.2	0.23
34	0.49	0.08
35	--	-0.58
36	1.1	1.1
37	0.08	0.84
38	0.08	-0.56

Notes:

1. Z-scores marked with asterisk (*) are regarded as unsatisfactory (i.e. $|z| \geq 3$) and the laboratories concerned should investigate their results.
2. Laboratories having a z-score in the range $2 < |z| < 3$ are encouraged to review their results.

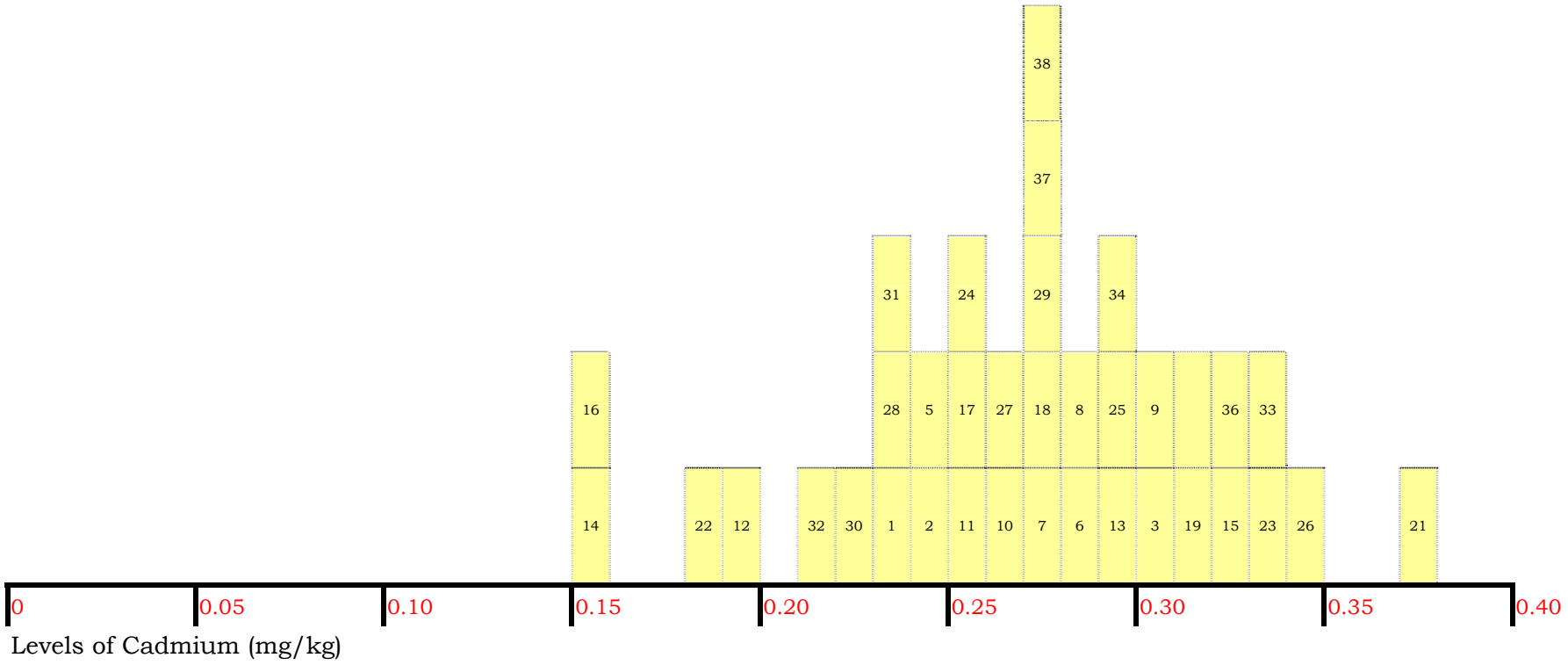

Figure 1 - Levels of cadmium: histogram of data as reported (data from Table 4)

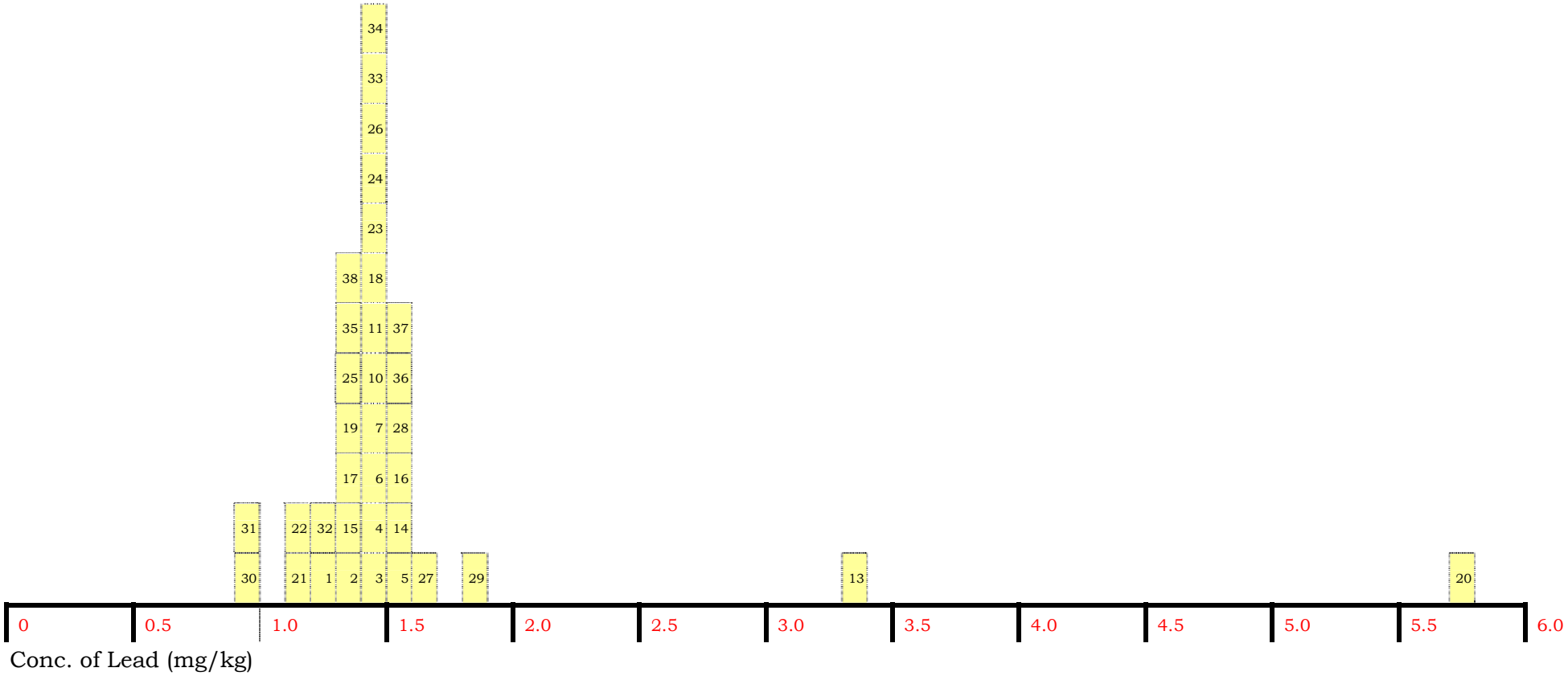

Figure 2 - Levels of lead: histogram of data as reported (data from Table 4)

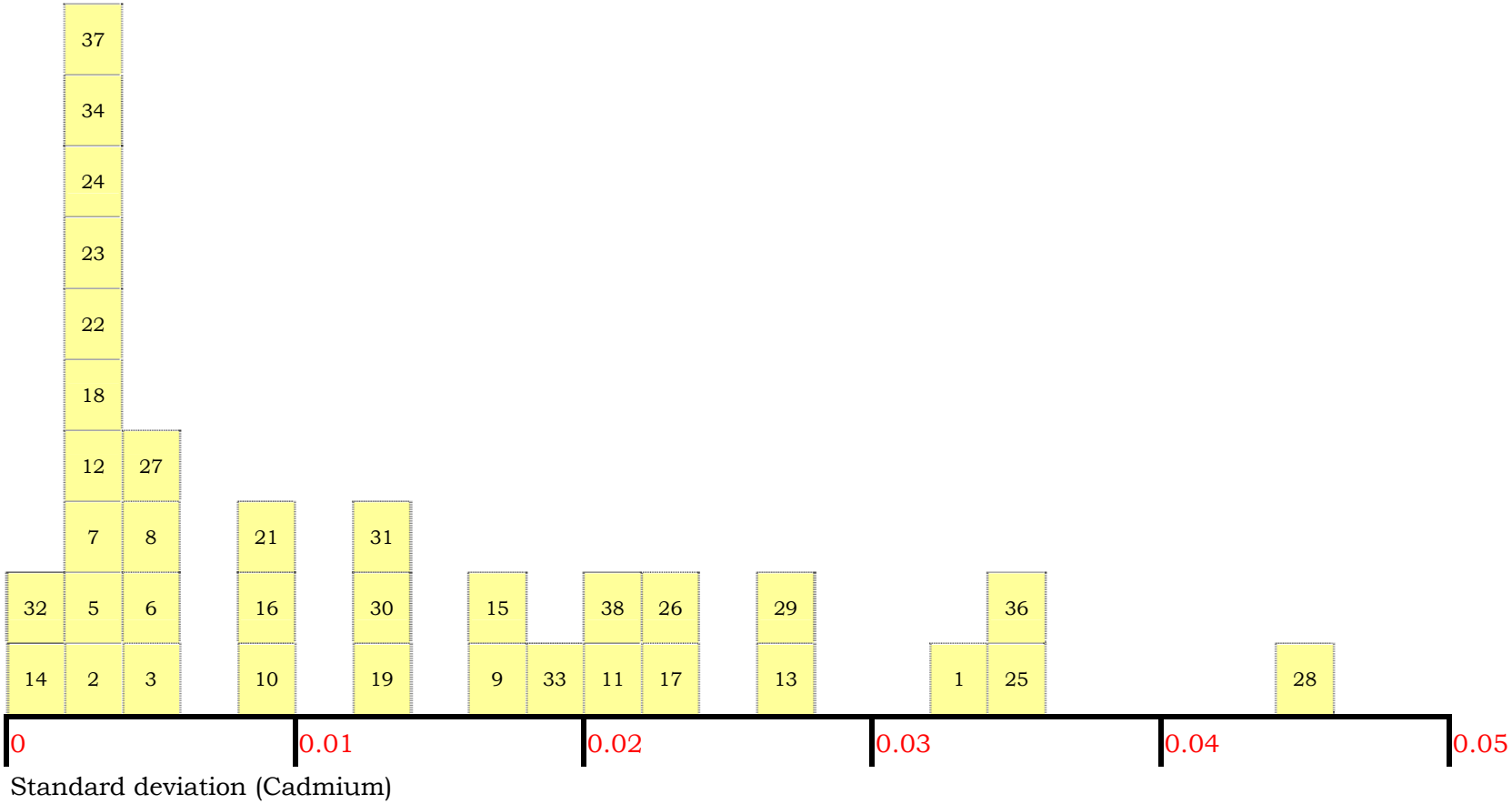

Figure 3 - Standard deviation (Cadmium): histogram of data as reported (data from Table 5)

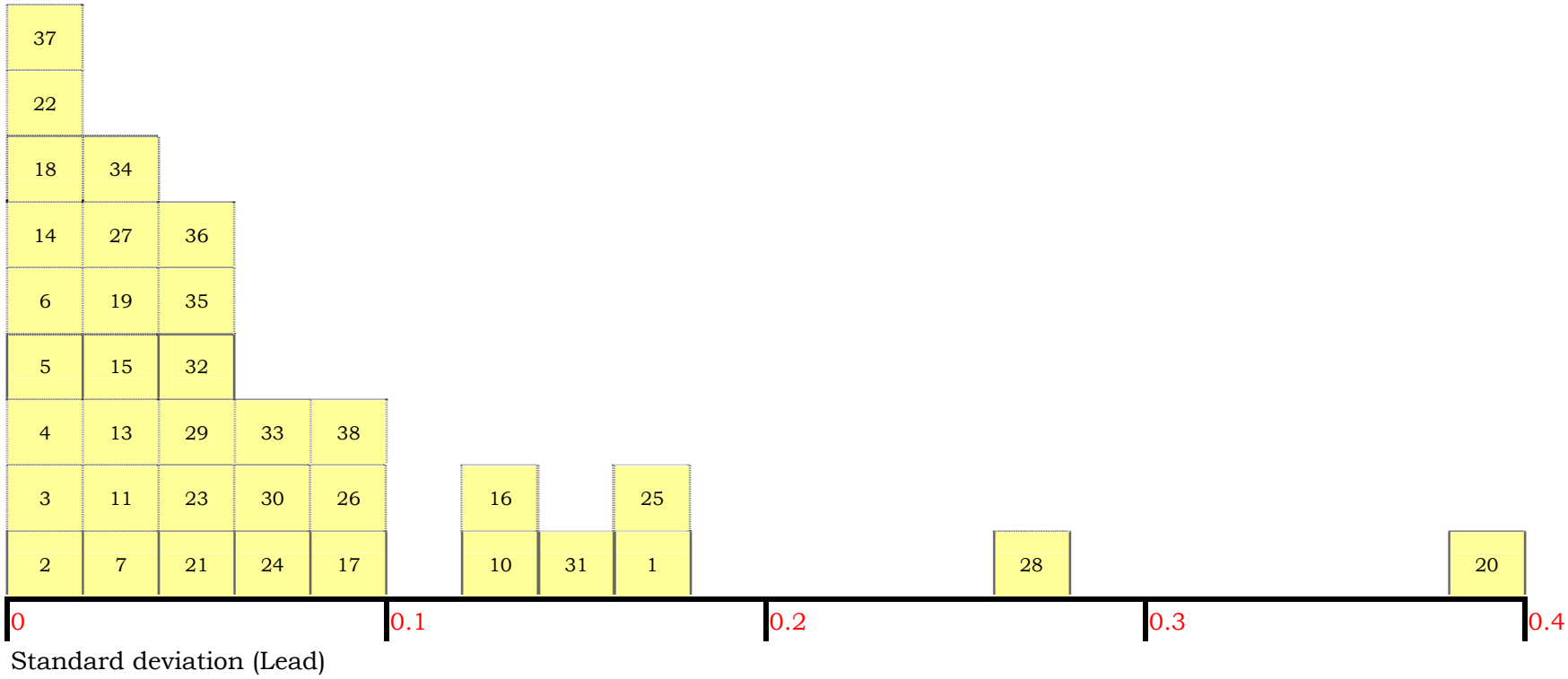

Figure 4 - Standard deviation (Lead): histogram of data as reported (data from Table 5)

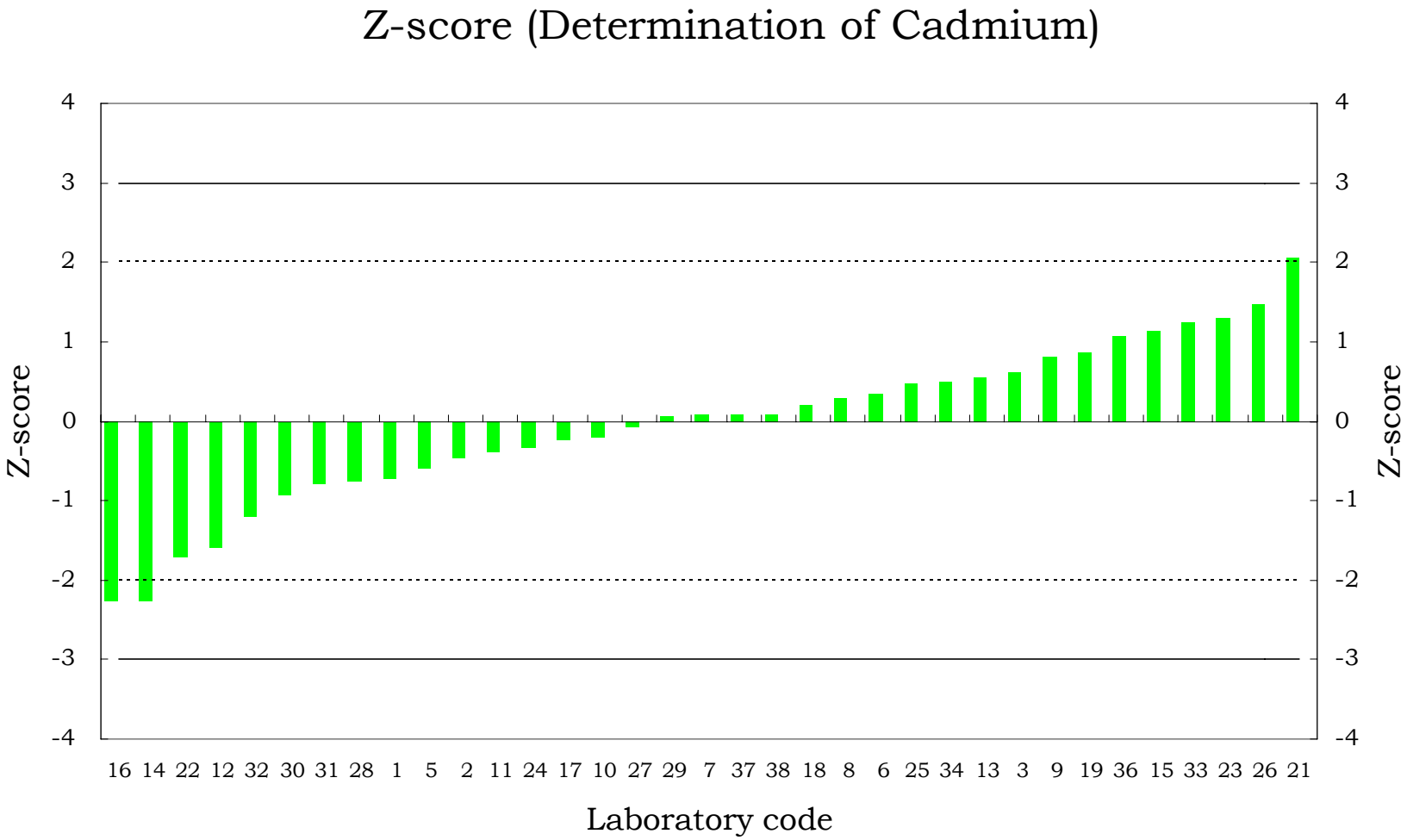

Figure 5 : Z-score (Determination of Cadmium)

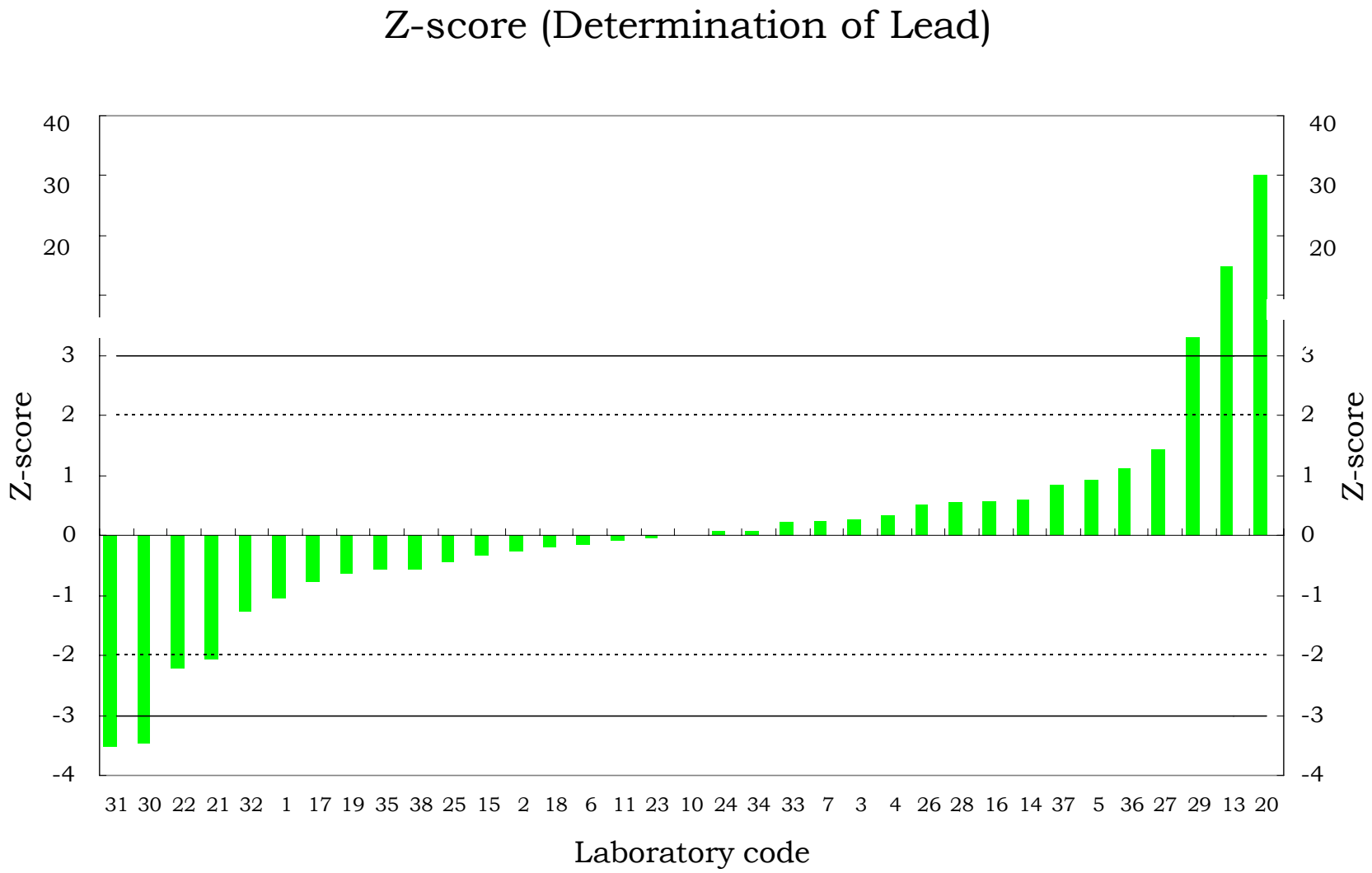

Figure 6 : Z-score (Determination of Lead)