



APMP-APLAC Joint Proficiency Testing Programme
(APLAC PT T100)
Toxic Elements (Lead and Cadmium) in Wheat Flour



**Asia Pacific Metrology Programme (APMP) -
Asia Pacific Laboratory Accreditation Cooperation (APLAC)
Joint Proficiency Testing Programme
<APLAC PT T100>
Toxic Elements (Lead and Cadmium) in Wheat Flour**

Final Report

Jointly coordinated by:

Korea Research Institute of Standards and Science (KRISS)
and
Korea Laboratory Accreditation Scheme (KOLAS)

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Summary of Results

1. This proficiency testing program (APLAC PT T100) was jointly organized by Asia-Pacific Metrology Programme (APMP) and Asia Pacific Laboratory Accreditation Corporation (APLAC) and jointly coordinated by Korea Research Institute of Standards and Sciences (KRIS), a member of APMP, and Korea Laboratory Accreditation Scheme (KOLAS), a member of the APLAC. The main objective of the program is to assist participating laboratories in demonstrating competence in the quantitative analysis of two hazardous elements, cadmium and lead, in wheat flour.
2. The design of the proficiency test followed the requirements in ISO/IEC 17043:2010 Conformity Assessment – General Requirements for Proficiency Testing [1] and the test results submitted by participants were processed in accordance with the statistical techniques outlined in ISO13528:2015 Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons [2].
3. A total of 95 laboratories from 44 economies were nominated, 91 out of 95 laboratories from 43 economies enrolled in the program and 88 of them returned their results (both cadmium and lead results). One laboratory submitted only cadmium result and reported ND (not detected) for lead result. One laboratory reported the results as a range not values. Participants were confidentially assigned with unique laboratory codes (T100-01 to T100-95) and the codes were used throughout the program.
4. The reference values certified by KRIS for cadmium and lead in the test sample were used as the assigned values for evaluating the performance of participants of this PT. The standard deviations for performance assessment were calculated using the Horwitz Equation [3]. The z-scores were used as numerical indicators to show participants' performance with respect to the assigned values in the program.
5. Performance of participants, in terms of z-scores, is summarized as follows:

Z-score	Number of Participants (Percentage)	
	Cadmium	Lead
$ z \leq 2.0$	80 (92 %)	72 (84 %)
$2.0 < z < 3.0$	4 (5 %)	5 (6 %)
$ z \geq 3.0$	3 (3 %)	9 (10 %)
Total:	87 (100 %)	86 (100 %)



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1. Introduction

- 1.1. Wheat (*Triticum aestivum* L.) is the most important crop worldwide, followed by coarse grains and rice. It is the basic food of eighty percent of the world population. Wheat crop covers the largest area in the world agricultural production. Wheat contains a number of elements as well as nutrients. The elements such as copper, zinc, iron, nickel and manganese are essential for our biological functions, but high concentrations of such elements are hazardous to our health. Wheat also may contain certain hazardous elements such as lead, cadmium, arsenic and mercury. Agricultural products like wheat are prone to be contaminated with toxic elements from fertilizer, contaminated agricultural soil and water. Many countries set food safety regulations limiting the amount of toxic elements in agricultural products. With increasing international trade of food and agricultural products, traceable measurements of hazardous elements in agricultural products have become one of the essential requirements for ensuring food safety. Codex Alimentarius Commission (CAC) also set maximum levels of contaminants in food and feed to protect public health. In the case of lead or cadmium in wheat the maximum level is 0.2 mg/kg [4].
- 1.2. With the aim of enhancing the quality and traceability of measurements in various economies of the Asia-Pacific region through a better regional scientific infrastructure, the Asia-Pacific Metrology Programme (APMP) and the Asia Pacific Laboratory Accreditation Cooperation (APLAC) agreed to strengthen bilateral cooperation. The Korea Research Institute of Standards and Sciences (KRISS), a member of APMP, and the Korea Laboratory Accreditation Scheme (KOLAS), a member of APLAC, have jointly proposed a PT scheme for the determination of toxic elements (lead and cadmium) in wheat flour. The purpose of this study is to demonstrate the capability of participating laboratories in measuring toxic elements in the test sample of wheat flour. A domestic PT round for Ministry of Food and Drug Safety (MFDS) in Korea was conducted in parallel with the APMP-APLAC Joint PT.
- 1.3. Reference values provided by KRISS for cadmium and lead in the test sample will be used as the assigned values for evaluating measurement results of participants. The relevant Calibration and Measurement Capabilities (CMCs) of KRISS are registered in the Key Comparison Data Base (KCDB) of the Comité International des Poids et Mesures (CIPM, International Committee for Weights and Measures) Mutual Recognition Arrangement (MRA). The use of reference values traceable to the International System of Units (SI, *Système international d'unités*) provided by



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National Metrology Institutes (NMIs) with appropriate CMCs as PT reference values at this and forthcoming APMP-APLAC Joint PTs will allow the rigorous evaluation of the accuracy of participants' results. It will enhance the quality of the PT programme and also help build the measurement capabilities of the participants through a better regional linkage between the NMIs and the analytical laboratories in the Asia-Pacific region.

- 1.4. A total of 91 laboratories from 43 economies enrolled in the program and 88 of them returned their results report form (TABLE I) on or before the final deadline (TABLE II). Participants were confidentially assigned with unique laboratory codes (T100-01 to T100-95) and the codes were used throughout the program.

2. Measurand

- 2.1. Mass fraction (mg/kg) on a dry mass basis of hazardous elements, cadmium and lead, in wheat flour.

3. Objectives

- 3.1. The aim of this study is to demonstrate the capability of participating laboratories in measuring the amount of lead and cadmium in the test sample of wheat flour by various analytical techniques.
- 3.2. The design of the proficiency test followed the requirements in ISO/IEC 17043:2010 Conformity Assessment – General Requirements for Proficiency Testing [1] and the test results submitted by participants were processed in accordance with the statistical techniques outlined in ISO13528:2015 Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons [2].

4. Test Material

- 4.1. The test material was prepared by KRISS. Approximately 22 kg of wheat flour produced by a local company was purchased from a local market in Korea. Appropriate amounts of lead and cadmium solutions were added into the wheat flour to make it into a paste form, and then mixed in a Teflon-coated mixing bowl over 4 hours. The mixed wheat flour paste was then frozen for 2 hours and then dried for over 130 hours using a freeze dryer (PVRFD 100R, Ilshin Lab, Korea) with capacity of 100 kg. The weight loss due to freeze-drying was about 13 %. Freeze-dried wheat flour was ground using a laboratory mill (Pulvurissette 14; Fritsch, Idar-Oberstein, Germany) with

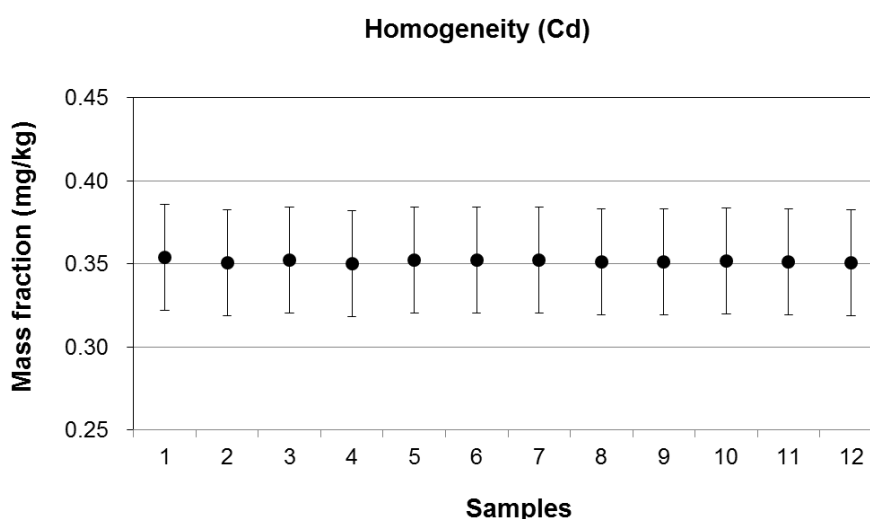


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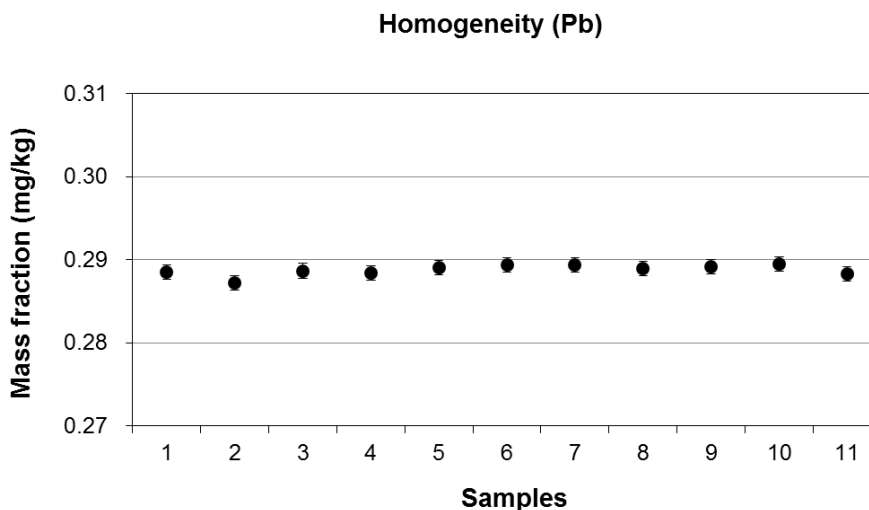
a 0.5 mm sieve ring. The speed of rotor was 12000 rpm. Dried wheat flour was sieved using a vibrating sifter (V/Sifter-141, Daega, Korea) to collect powder with limited range of particle size, 50 μm ~ 250 μm . It was homogenized with a V-blender (Daega Powder, Korea) for over 10 hours and then bottled into pre-cleaned 60 mL amber bottles in 20 g per unit. The sample bottles were sealed and then sterilized by irradiation of ^{60}Co gamma rays at a dose of about 25 kGy. Sample bottles were stored at room temperature prior to distribution or use.

- 4.2. The homogeneity study of the proficiency test sample was carried out using Isotope dilution Inductively Coupled Plasma Mass Spectrometry (ID-ICP/MS) analysis after microwave digestion of subsamples. More than ten bottles were taken with even interval following the bottling order from the sample batch. One subsample from each bottle with a minimum sampling size of 0.5 g was analysed. The relative standard deviations of Cd and Pb contents due to between-sample inhomogeneity were less than 0.29 % and 0.23 %, respectively. The between-samples standard deviation (s_s) was compared with the standard deviation for the proficiency assessment (σ_{pt}) and found to meet the requirement, $s_s \leq 0.3\sigma_{pt}$, both for Cd and Pb. Therefore, the samples may be considered to be adequately homogeneous based on ISO 13528 [2]. The plots showing the results of homogeneity assessments for analytes (Cd and Pb) are shown in the following figures.





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- 4.3. The contents of non-volatile elements for food materials in a dried powder form sterilized with gamma ray irradiation are generally known to be stable for more than 5 years at room temperature. The stability assessment on the similar batch of rice powder CRM also demonstrated excellent stability of non-volatile elements in dried food materials. Therefore, the stability of the proficiency test sample was expected to be satisfactory for the purpose of present proficiency testing based on the previous stability studies of KRIS.
- 4.4. One bottle containing about 20 g of the wheat flour was dispatched to each participant by express mail on 6 July 2015. The document of “Sample Receipt Form” was sent to participants via emails at the time of sample dispatch. The copies of the documents including “Instruction to Laboratory”, “Sample Receipt Form”, and “Result Report Form” are given in APPENDICES I-III.
- 4.5. Participants were asked to check the physical condition of the sample upon receipt and promptly acknowledge receipt and condition of the sample by returning the “Sample Receipt Form” to aplacpt.inorg@kriss.re.kr. Replacement would be arranged if any defect was detected. 83 laboratories returned their sample receipt form. Extra samples were sent to one participant (Lab code T100-72) due to delivery problem. The samples were returned from participant (Lab code T100-64) without any notice.



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5. Test and Reporting

- 5.1. Participants were requested to determine the mass fraction (in mg/kg) of cadmium and lead on a dry mass basis in the sample with their preferred methods, which should be consistent with their routine procedure. The organizer recommended following the guidelines for sample preparation and dry-mass correction.
- (i) Sample handling in a laboratory with well-controlled humidity and temperature is recommended. Any kind of contamination should be avoided.
 - (ii) Sampling for the analysis should be carried out at the same time as sampling for the dry-mass correction.
 - (iii) Digest the wheat flour material completely, if the method used requires digestion.
 - (iv) The proficiency test sample should be mixed thoroughly before taking an aliquot of sample from a bottle.
 - (v) The analysis should be conducted with a recommended sample size of at least 0.5 g.
 - (vi) Participants should also carry out the dry mass correction.
 - (vii) Sampling for the dry mass correction should be carried out at the same time as the sampling for analysis.
 - (viii) At least three separate portions from a sample bottle (with a recommended sample size of about 0.5 g for each portion) should be taken as dry mass correction samples and placed over P_2O_5 in a desiccator at room temperature for 7 days (168 hours).
 - (ix) Calculate the moisture content from the mass change observed in the three aliquots and use it for dry-mass correction.
- 5.2. Participants were recommended to perform at least triplicate measurements and report the mean and associated measurement uncertainty for each analyte as specified in the Result Report.
- 5.3. Participants were requested to report the mean value of at least three independent measurements, its associated expanded uncertainty with 95 % level of confidence and information about the method of analysis, such as digestion technique, calibration method and analytical instrument etc. on the “Result Report” provided and submit the completed form to the coordinator electronically to aplacpt.inorg@kriss.re.kr before the deadline on 4 September 2015.



6. Assigned Values for Proficiency Assessment

- 6.1. The reference values certified by KRISS were used as the assigned values, proficiency test reference value (PTRV). KRISS used a robust primary method, the double isotope dilution ICP/MS, to determine the reference values and their associated uncertainties. This is in accordance with the ISO/IEC 17043 recommendations on the determination of assigned values for proficiency testing schemes.
- 6.2. The assigned values (x), standard uncertainties (u_x), coverage factor (k) and expanded uncertainties (U_x) [5] are summarized as follows:

Analyte	Assigned value (x) (mg/kg)	Standard uncertainty (u_x) (mg/kg)	Coverage factor (k)	Expanded uncertainty (U_x) (mg/kg)
Cadmium	0.352	0.011	1.960	0.022
Lead	0.2888	0.0011	2.007	0.0022

The standard uncertainty was estimated by combining the standard deviations due to between-sample variation (0.29 % and 0.23 % for Cd and Pb) and uncertainty contribution from the systematic effects (3.19 % and 0.30 % for Cd and Pb).

- 6.3. The level of cadmium and lead in the wheat flour is similar to the maximum level (0.2 mg/kg) set by Codex [4].

7. Evaluation of Performance of Participants

- 7.1. Participants' performance was assessed using the z-score, which is calculated as follows:

$$z = \frac{x_i - x_{pt}}{\sigma_{pt}}$$

where x_i = the reported result of the individual participant
 x_{pt} = the assigned value
 σ_{pt} = the standard deviation for proficiency assessment estimated from the Horwitz Equation
 $[\sigma = 0.02c^{0.8495}]$, where c is the assigned value of the analyte expressed as a dimensionless mass ratio (e.g. 1 mg/kg = 1 ppm = 10^{-6})

- 7.2. The z-score is commonly interpreted as:



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- (i) $|z| \leq 2.0$ Satisfactory
- (ii) $2.0 < |z| < 3.0$ Questionable
- (iii) $|z| \geq 3.0$ Unsatisfactory

- 7.3. Laboratories having $|z| \geq 3.0$ should thoroughly investigate their results for the discrepancy and those having z-scores in the range $2.0 < |z| < 3.0$ are also encouraged to review their results.
- 7.4. Participants were requested to review the interim report and confirm the correctness of the data transcription before 22 January 2016. Fifty five participants reviewed their results and confirmed that there was no transcription error.

8. Results and Discussion

- 8.1. Reported test results and the technical information of methods used by participants are presents in Tables III to IX.
- 8.2. Eighty seven cadmium results and eighty six lead results were received. Eighty six out of all participants submitted both results.



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8.3. An overview of participants' results is summarized in the following table:

	Cadmium	Lead
No. of Results	87	86
Assigned value	0.352 mg/kg	0.2888 mg/kg
Median of Participants' Results	0.342 mg/kg	0.280 mg/kg
Robust Average	0.341 mg/kg	0.289 mg/kg
Average of Participants' Results	0.347 mg/kg	0.310 mg/kg
Standard Deviation of Participants' Results	0.087 mg/kg	0.133 mg/kg
Relative Standard Deviation of Participants' Results	25.2 %	42.9 %
Robust Standard Deviation	0.054 mg/kg	0.062 mg/kg
Standard Deviation Estimated from the Horwitz Equation	0.066 mg/kg	0.056 mg/kg
Difference of the average from PTRV	-0.005 mg/kg	0.021 mg/kg
Relative Difference of the Median from PTRV	-2.8 %	-3.0 %
Relative Difference of the average from PTRV	-1.5 %	7.4 %

8.4. Proficiency test reference values (PTRV), the certified values provided by KRISS, for cadmium and lead in the sample were used as the assigned values to evaluate the performance of the participants. We observed a very small difference of the average of the participants' results with the assigned values. The medians of participants are 2.8 % and 3 % lower and robust averages are 3 % lower and 0.07 % higher than the corresponding RV for cadmium and lead, respectively. The robust average is quite similar to assigned value for lead, indicating the competence of participants on the quantitative analysis of lead. For cadmium and lead, the standard deviation obtained from the participants is much larger than σ_H (obtained from the model of Horwitz based on ISO 13528), which is general model adopted for the estimation of the reproducibility of analytical methods. This might be due to the scattering of the participants' results. The robust standard deviation (obtained from robust analysis based on ISO 13528) is quite similar to σ_H , indicating good fitting



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of this study with the general model of Horwitz. The z-scores are calculated by adopting σ_H as the expected standard deviation of this study, which was announced in the study protocol. In fact, Horwitz Equation is generally accepted in many proficiency testing schemes as defining fitness-for-purpose especially for food analysis.

- 8.5. Participants' z-scores, zeta scores, E_n scores and reported results are presented in TABLEs III, IV and V. The performance of the participating laboratories is assessed using z-score only. The pattern of z-score distribution in ascending order, for all participants, is shown in FIGUREs I and II. The results with z-scores marked bold with blue colour or red colour were considered as questionable (i.e. $2.0 < |z| < 3.0$) or unsatisfactory (i.e. $|z| \geq 3.0$), respectively. The results with $|\zeta| \geq 3.0$ or $|E_n| > 1.0$ are marked with bold. The standard uncertainty and expanded uncertainty reported from each participant were used to derive zeta-score and E_n score just for information. The calculation of zeta-score and E_n score of the reported results were calculated following ISO 13528. The zeta scores and E_n scores are effective for evaluation the reported results considering their estimated standard uncertainties and expanded uncertainties because the reported results were not used to calculate the reference value here. It can be seen that more participants got "questionable or unsatisfactory results" when the results were assessed by zeta-scores and E_n scores. This fact indicates that the present PT was required for the participants to improve their uncertainty evaluation.
- 8.6. The comparisons between the assigned values and the participants' reported results with associated expanded uncertainties, if provided, are also presented in FIGUREs III and IV.
- 8.7. Performance of participants, in terms of z-scores, is summarized as follows:

z-score	Number of Participants (Percentage)	
	Cadmium	Lead
$ z \leq 2.0$	80 (92 %)	72 (84 %)
$2.0 < z < 3.0$	4 (5%)	5 (6 %)
$ z \geq 3.0$	3 (3 %)	9 (10 %)
Total:	87 (100 %)	86 (100 %)



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In brief, sixty eight participants (78 %) reported satisfactory results with $|z| \leq 2.0$ for both analytes. Two participants (Lab Code: T100-72 and T100-88) were identified as having unsatisfactory results with $|z| \geq 3.0$ for both analytes and eight participants were found to have unsatisfactory results for one of the analytes.

- 8.8. Youden graphs showing the results distribution among the participants is shown in Figures V, VI, VII, IX, XI, XIII, and XV. X axis and Y axis indicate z-scores for Cd and Pb. Each point represents a pair of results from a single laboratory. The different coloured symbols identify the different instrumental methods, the use of internal standard, digestion technique, correction for recovery, method validation. If results from different laboratories vary entirely because of random error, one would expect approximately equal number of points in each quadrant. If systematic errors were the main cause of the variation, one would expect that laboratories with a high value for one analyte would also tend to obtain a high value for another analyte. This would lead to a predominance of points in the lower left and upper right quadrants.
- 8.9. Figures VIII, X, XII, XIV and XVI are the results for Cd and Pb, which are sorted according to the accreditation for the test, instrumental methods used, use of internal standard, digestion technique, recovery correction and method validation.
- 8.10. Instrumental methods:

As shown in Tables VIII, Table IX, Figures VII and VIII, all participants employed ICP-MS, ICP-OES (ICP-AES), Flame AAS or Graphite AAS for measurements. Over one-third of the participants (38 participants) used ICP-MS to quantify the levels of both analytes. Twelve participants used ICP-OES (ICP-AES) to quantify the analytes. Seven participants and twenty six participants employed Flame AAS and Graphite AAS to quantify both analytes. Some large relative deviations from PTRV for Pb or Cd were observed when Flame AAS or Graphite AAS were used compared to ICP-MS. The Youden plot and results sorted according to the instrumental methods are shown in Figures VII and VIII respectively.

- 8.11. The use of internal standard:
- Forty one participants (47 %) applied internal standard to quantify the analytes. Thirty four (83 %) out of forty one participants were found having satisfactory results with $|z| \leq 2.0$ on both analytes. The Youden plot and results sorted according to the use of internal standard are shown in Figures IX and X. As shown in Figure IX, it is found that laboratories have $|z| \leq 2.0$ mostly when



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participants used an internal standard to quantify the analytes. Some large relative deviations from PTRV for Pb or Cd were observed when an internal standard was not used.

8.12. Digestion technique:

Fifty nine participants (about 68 %) employed Microwave-assisted digestion to dissolve samples. Thirteen participants and twelve participants employed wet digestion to dissolve samples to analyse Cd and Pb respectively. Fourteen participants dissolved samples by dry ashing to analyse both analytes. The Youden plot and results sorted according to their digestion technique are shown in Figures XI and XII.

8.13. Correction for recovery:

Only sixteen participants (18 %) corrected their results for recovery. The Youden plot and results sorted according to their correction for recovery are shown in Figures XIII and XIV.

8.14. Method validation:

Sixty two (about 71 %) participants claimed their methods are validated for both or one of analytes. Ten out of sixty two (16 %) from laboratories which used a validated method were identified as questionable or unsatisfactory, while there are five out of nineteen (26 %) data from laboratories that used non-validated methods. Youden plot and results sorted according to the use of validated method are shown in Figures XV and XVI.

9. Additional Observations

- 9.1. One participant (Lab code: T100-06) submitted only cadmium result and reported ND (not detected) for lead result. The lead result was not included in evaluation of performance of participants.
- 9.2. One participant (Lab code: T100-75) reported the results with range not values. Resubmission of results with value was requested but participant wants to keep the result with range of value. The results were not included in evaluation of performance of participants.
- 9.3. One participant (Lab code: T100-41) reported that results cannot be submitted due to problems with equipment.
- 9.4. Test sample delivered to one laboratory (Lab code: T100-64) was returned back to coordinator. The sample status was informed to laboratory for successful delivery when the sample was arrived at post office in its country. Any response from laboratory was not arrived.



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9.5. The 83 of 91 participants returned sample receipt form and 88 submitted result report form.

10. Remarks

- 10.1. Contributions from all accreditation bodies, participants to this program are gratefully noted. Special thanks are also extended to APLAC PT Committee, APMP-APLAC Joint PT Working Group Members, and the KOLAS for their support to the program.
- 10.2. Further comments on the report should be promptly forwarded by email to the following:
Dr. Sook Heun Kim, aplacpt.inorg@kriss.re.kr
- 10.3. Use of this report by participants shall only be allowed with the written permission of the program's organizers.

11. References

- [1] ISO/IEC 17043:2010. Conformity Assessment – General Requirements for Proficiency Testing, International Organization for Standardization, Geneva, Switzerland, 2010.
- [2] ISO 13528:2015, Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons, International Organization for Standardization, Geneva, Switzerland, 2015.
- [3] Horwitz, W. Evaluation of analytical methods used for regulations of food and drugs, *Anal. Chem.*, **1982**, 54: 67A-76A.
- [4] Codex Standard 193-1995, Codex General Standard for Contaminants and Toxins in Food and Feed, Codex Alimentarius Commission.
- [5] JCGM100 (2008) Evaluation of measurement data - Guide to the expression of uncertainty in measurement (GUM:1995 with minor corrections), 1st edition (corrected 2010), BIPM/IEC/IFCC/ILAC/ISO/IUPAC/OIML (published by JCGM).
http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf



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Table I: Geographical distribution of participants

No.	ECONOMIES	PARTICIPANTS ENROLLED	RETURNED RESULTS
1	UAE	1	1
2	Viet Nam	4	4
3	Hong Kong	4	4
4	Israel	2	2
5	China	2	2
6	Taiwan	4	4
7	Serbia, Republic of	2	1
8	USA	2	2
9	Singapore	1	1
10	Russia	4	3
11	France	2	2
12	Austria	2	2
13	India	4	4
14	Canada	3	3
15	Slovenia	2	2
16	Finland	2	2
17	Lithuania	2	2
18	Greece	2	2
19	Croatia	2	2
20	Denmark	1	1
21	Philippine	3	3
22	Japan	3	3
23	Brunei Darussalam	1	1
24	Hungary	1	1
25	Slovakia	2	2
26	Romania	2	2
27	Korea	1	0
28	Indonesia	3	3
29	Bosnia and Herzegovina	2	2
30	New Zealand	3	3
31	Czech Republic	2	2
32	Ireland	1	1
33	Malaysia	3	3
34	United Kingdom	2	2
35	Switzerland	2	2
36	Ethiopia	2	2
37	Norway	1	1
38	Turkey	2	2
39	Portugal	1	1
40	Thailand	1	1
41	Bangladesh	3	3
42	Sweden	1	1
43	Pakistan	1	1
Total:		91	88



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Table II: Program timeline

PHASE	TIME SCHEDULE
Preparation of sample	July 2014
Homogeneity test	December 2014
Call for participants	May 2015
Dispatch of samples	6 July 2015
Deadline for submission of results	4 September 2015
Statistical analysis of results	October 2015
Issue of interim report	22 December 2015
Issue of final report	October 2016

Table III: Participants' z-scores, zeta-scores, and E_n score for cadmium and lead

Lab Code	Cd			Pb		
	z-score	ζ -score	E_n score	z-score	ζ -score	E_n score
T100-01	0.35	1.84	0.92	0.24	2.16	1.00
T100-02	-0.12	-0.44	-0.22	0.34	0.94	0.47
T100-03	1.18	5.41	2.68	1.64	6.06	3.03
T100-04	-1.55	-3.67	-1.84	0.38	0.66	0.33
T100-05	-1.24	-1.77	-0.89	-0.70	-0.86	-0.43
T100-06	2.40	---	---	---	---	---
T100-07	0.02	0.04	0.02	0.11	0.21	0.12
T100-08	-1.73	-10.32	-5.16	1.90	54.39	26.63
T100-09	-0.49	-1.97	-1.01	-0.34	-1.44	-0.75
T100-10	-0.33	-0.22	---	-0.05	-0.03	---
T100-11	-0.05	-0.15	-0.04	-0.23	-0.98	-0.22
T100-12	-0.33	-1.09	-0.59	0.02	0.08	0.04
T100-13	-1.11	-0.86	-0.43	-0.89	-0.35	-0.18
T100-14	-3.16	-8.46	-4.23	-1.74	-2.02	-1.01
T100-15	-1.06	0.00	0.00	-0.62	0.00	0.00
T100-16	-0.33	-1.97	-0.98	0.79	9.16	4.63
T100-17	0.36	1.38	0.69	0.08	0.32	0.16
T100-18	-2.99	-11.57	-5.66	0.94	2.17	1.09
T100-19	-0.18	-0.53	-0.09	-0.70	-34.70	-0.31
T100-20	-1.85	-6.85	-3.32	3.97	15.75	7.48
T100-21	0.00	0.00	0.00	2.21	2.80	1.40
T100-22	-0.23	-1.33	-0.66	-0.52	-13.12	-6.31
T100-23	0.47	1.41	1.21	0.09	0.37	0.71
T100-24	-0.03	-0.04	-0.02	-0.52	-0.76	-0.37
T100-25	-0.18	-0.81	-0.32	0.20	0.56	0.28
T100-26	-0.18	---	-0.50	0.74	---	4.02
T100-27	-0.03	-0.04	-0.02	-0.39	-0.59	-0.29
T100-28	0.16	0.72	0.36	-0.08	-0.17	-0.09
T100-29	-0.18	-0.30	-0.15	5.00	4.28	2.14
T100-30	0.09	0.20	0.10	0.25	0.33	0.17
T100-31	0.02	0.08	0.04	-0.21	-2.30	-1.15
T100-32	N/A	N/A	N/A	N/A	N/A	N/A
T100-33	0.20	0.31	0.16	-0.01	-0.03	-0.01
T100-34	0.65	0.59	0.30	-0.57	-0.69	-0.35



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Lab Code	Cd			Pb		
	z-score	ζ -score	E_n score	z-score	ζ -score	E_n score
T100-35	-0.26	-1.42	-0.71	-0.71	-6.22	-3.71
T100-36	-1.23	-2.15	-1.08	-0.50	-1.63	-0.79
T100-37	0.32	0.17	0.09	-1.13	-1.65	-0.82
T100-38	-0.15	-0.47	-0.24	0.20	0.70	0.35
T100-39	-0.14	-0.63	-0.31	0.02	0.04	0.02
T100-40	-2.29	-13.24	---	-3.19	-34.73	---
T100-41	—	—	—	—	—	—
T100-42	0.21	1.20	0.60	1.56	2.46	1.23
T100-43	-0.79	---	---	0.02	---	---
T100-44	-1.40	-0.57	-0.29	-1.59	-2.96	-1.48
T100-45	N/A	N/A	N/A	N/A	N/A	N/A
T100-46	-0.42	-1.44	-0.75	0.13	1.74	0.70
T100-47	-1.68	-6.09	-3.05	8.68	39.17	19.58
T100-48	-0.30	-0.69	-0.35	0.88	0.99	0.49
T100-49	-0.02	-0.02	-0.01	0.27	0.33	0.17
T100-50	0.12	0.54	0.22	-0.34	-1.87	-0.67
T100-51	-0.06	-0.07	-0.03	-0.16	-0.17	-0.09
T100-52	-0.94	---	---	-0.52	---	---
T100-53	-0.17	-0.65	-0.32	-0.16	-0.29	-0.15
T100-54	N/A	N/A	N/A	N/A	N/A	N/A
T100-55	-0.32	-0.82	-0.41	-0.25	-0.37	-0.19
T100-56	-0.18	-0.38	-0.19	1.10	1.22	0.61
T100-57	-0.73	-1.50	-0.75	-0.05	-0.14	-0.07
T100-58	1.20	3.46	1.73	-2.22	-3.09	-1.38
T100-59	-0.08	-0.09	-0.05	-0.73	-1.10	-0.55
T100-60	N/A	N/A	N/A	N/A	N/A	N/A
T100-61	-0.49	---	---	-0.88	---	---
T100-62	-0.97	-3.76	-1.88	-0.73	-5.76	-2.88
T100-63	0.29	0.36	0.18	1.37	1.47	0.73
T100-64	—	—	—	—	—	—
T100-65	-0.11	-0.23	-0.11	-0.70	-10.86	-0.57
T100-66	0.73	2.58	1.29	0.38	0.96	0.48
T100-67	-0.29	-0.80	-0.49	-0.84	-5.28	-3.55
T100-68	-0.64	---	-0.92	1.46	---	1.35
T100-69	0.11	---	0.20	-0.28	---	-0.56



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Lab Code	Cd			Pb		
	z-score	ζ -score	E_n score	z-score	ζ -score	E_n score
T100-70	-0.49	-1.21	-0.60	-1.06	-3.91	-1.95
T100-71	—	—	—	—	—	—
T100-72	7.41	---	---	14.92	---	---
T100-73	1.12	3.49	1.75	4.31	7.55	3.77
T100-74	-1.24	-0.82	-0.41	-0.88	-0.41	-0.24
T100-75	---	---	---	---	---	---
T100-76	-0.64	-2.36	-1.18	4.51	41.18	20.59
T100-77	0.20	0.40	0.20	-0.54	-2.70	-1.35
T100-78	0.27	0.56	0.28	2.36	2.62	1.31
T100-79	0.24	1.08	0.54	-2.33	-15.54	-7.75
T100-80	0.12	0.38	0.19	-0.70	-6.36	-3.18
T100-81	1.79	5.40	2.69	5.23	18.86	9.46
T100-82	-0.49	-1.65	-0.82	-0.88	-4.05	-2.02
T100-83	0.88	3.90	1.56	-0.34	-1.87	-0.93
T100-84	-0.14	-0.20	-0.10	-0.07	-0.20	-0.10
T100-85	1.15	3.33	1.66	-0.16	-0.42	-0.21
T100-86	0.73	1.16	0.58	-1.59	-2.22	-1.27
T100-87	-0.24	-0.45	-0.23	-0.16	-0.16	-0.08
T100-88	4.22	---	---	-3.39	---	---
T100-89	-0.94	-2.43	-1.13	0.20	0.34	0.19
T100-90	-1.27	---	---	-0.52	---	---
T100-91	-0.23	-0.32	-0.16	-0.61	-0.88	-0.44
T100-92	0.96	---	2.42	-0.89	---	-4.44
T100-93	0.62	3.56	1.78	-1.20	-9.01	-4.51
T100-94	2.25	1.83	---	2.00	1.85	---
T100-95	-1.24	-2.81	-1.41	2.18	2.96	1.48

“—” The registered participant did not submit the results.

“---” Data or information was not provided.

N/A: nominated but not registered laboratory

* Lab T100-75 reported the range of value.



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Table IV: Participants' results, reported uncertainties, and laboratory accreditation status for cadmium

Lab Code	Moisture content (%) of the sample	Mass fraction of cadmium (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor, <i>k</i> (95% level of confidence)	Expanded uncertainty (mg/kg)	Laboratory accreditation (Yes/No)
T100-01	0.99	0.375	0.006	2	0.012	Yes
T100-02	1.21	0.344	0.0143	2	0.0286	Yes
T100-03	1.2	0.43	0.0093	2	0.019	Yes
T100-04	2.65	0.25	0.0255	2.0	0.051	Yes
T100-05	2.8	0.27	0.045	2	0.09	Yes
T100-06	8.10	0.51	---	---	---	No
T100-07	2.2	0.353	0.02	2	0.04	Yes
T100-08	---	0.2380	0.0010	2	0.0020	No
T100-09	1.96	0.32	0.012	2	0.023	Yes
T100-10	3.7	0.330	0.099	---	---	Yes
T100-11	---	0.349	0.017	4.303	0.072	Yes
T100-12	0.5	0.33	0.017	2	0.03	Yes
T100-13	99.34	0.279	0.0846	2	0.1692	Yes
T100-14	2.74	0.144	0.022	2	0.044	Yes
T100-15	0.07	0.282	99000	2	197000	Yes
T100-16	1.5	0.33	0.002	2	0.0046	Yes
T100-17	4.66	0.376	0.0134	2	0.0268	Yes
T100-18	4.80	0.155	0.013	2	0.027	Yes
T100-19	---	0.34	0.020	2	0.136	Yes
T100-20	0.098	0.23	0.014	2	0.0295	Yes
T100-21	1.83	0.352	0.0415	2	0.083	Yes
T100-22	0.049	0.337	0.0025	2	0.005	No
T100-23	0.9896	0.383	0.019	2.57	0.013	No
T100-24	1.8	0.35	0.056	2	0.11	Yes
T100-25	11.4	0.34	0.01	1.99	0.03	Yes
T100-26	---	0.34	---	---	0.01	Yes
T100-27	1.46	0.350	0.045	2	0.091	Yes
T100-28	1.503	0.3627	0.010	2	0.020	Yes
T100-29	3.21	0.340	0.038	2	0.075	Yes
T100-30	0.9	0.358	0.0287	2	0.0573	No
T100-31	1.5	0.353	0.007	2	0.014	No



Lab Code	Moisture content (%) of the sample	Mass fraction of cadmium (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor, k (95% level of confidence)	Expanded uncertainty (mg/kg)	Laboratory accreditation (Yes/No)
T100-32	N/A	N/A	N/A	N/A	N/A	Yes
T100-33	Results are NOT corrected for dry mass	0.365	0.04	2	0.08	Yes
T100-34	3	0.395	0.072	2	0.144	Yes
T100-35	2.86	0.335	0.0048	2	0.0096	Yes
T100-36	0.08	0.271	0.036	2	0.072	Yes
T100-37	10.8	0.373	0.121	2	0.242	Yes
T100-38	0.64	0.342	0.018	2	0.036	Yes
T100-39	1.49	0.343	0.00923	2	0.0185	Yes
T100-40	0.89	0.201	± 0.003	---	---	No
T100-41	—	—	—	—	—	Yes
T100-42	2.78	0.365536	0.002502	2	0.005004	Yes
T100-43	---	0.30	---	---	---	Yes
T100-44	3	0.26	0.16	2	0.32	Yes
T100-45	N/A	N/A	N/A	N/A	N/A	Yes
T100-46	0.727	0.324	0.016	2	0.03	Yes
T100-47	2.18	0.2411	0.0145	2	0.0290	Yes
T100-48	---	0.3324	0.0261	2	0.0521	Yes
T100-49	0	0.351	0.063	2	0.126	Yes
T100-50	9.4	0.36	0.01	2.8	0.028	Yes
T100-51	1	0.348	0.057	2	0.114	Yes
T100-52	3.59	0.29	---	---	---	Yes
T100-53	1.21	0.341	0.013	2	0.026	Yes
T100-54	N/A	N/A	N/A	N/A	N/A	Yes
T100-55	---	0.331	0.023	2	0.046	Yes
T100-56	1.9	0.34	0.03	2	0.06	Yes
T100-57	99.87	0.304	0.03	2	0.06	Yes
T100-58	7.32	0.431	0.02	0.01	0.04	Yes
T100-59	0.1	0.347	0.052	2	0.104	Yes
T100-60	N/A	N/A	N/A	N/A	N/A	Yes
T100-61	1.34	0.32	---	---	---	Yes
T100-62	2.82	0.288	0.013	2	0.026	Yes



Lab Code	Moisture content (%) of the sample	Mass fraction of cadmium (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor, <i>k</i> (95% level of confidence)	Expanded uncertainty (mg/kg)	Laboratory accreditation(Yes/No)
T100-63	2.48	0.371	0.052	2	0.104	Yes
T100-64	—	—	—	—	—	Yes
T100-65	1.56	0.345	0.029	2	0.059	No
T100-66	5.2	0.40	0.015	2	0.03	Yes
T100-67	---	0.333	0.021	2	0.032	Yes
T100-68	0.01	0.31	±0.02	2.0	0.04	Yes
T100-69	1.7	0.359	---	---	0.028	Yes
T100-70	0.66	0.32	0.024	2	0.049	Yes
T100-71	—	—	—	—	—	Yes
T100-72	0.63	0.84	---	2	---	No
T100-73	1.82	0.426	0.0181	2	0.0362	Yes
T100-74	2.3	0.27	0.10	2	0.2	Yes
T100-75	Not calculated	<2.4	---	---	---	Yes
T100-76	1.63	0.31	0.014	2	0.028	Yes
T100-77	2.275	0.365	0.031	2	0.062	Yes
T100-78	1.60	0.37	0.03	2	0.06	Yes
T100-79	2.68	0.368	0.0100	2.0	0.0200	Yes
T100-80	2.4	0.36	0.018	2	0.035	Yes
T100-81	1.72	0.47	0.0189	2	0.0379	Yes
T100-82	Not Done	0.32	0.016	2	0.032	Under
T100-83	1.58	0.41	0.01	2	0.03	Yes
T100-84	1.83	0.343	0.044	2	0.087	Yes
T100-85	0.99	0.428	0.020	2	0.040	Yes
T100-86	0.00 % dried in silica gel	0.4	0.04	2	0.08	Yes
T100-87	0.25	0.336	0.034	2	0.067	Yes
T100-88	Not Done	0.63	---	---	---	No
T100-89	3.29	0.29	0.023	1.96	0.05	Yes
T100-90	---	0.268	---	---	---	Yes
T100-91	1.15	0.337	0.0452	2	0.0905	Yes
T100-92	---	0.415	---	---	0.014	Yes
T100-93	1.35	0.393	0.00338	2	0.00676	Yes



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Lab Code	Moisture content (%) of the sample	Mass fraction of cadmium (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor, k (95% level of confidence)	Expanded uncertainty (mg/kg)	Laboratory accreditation (Yes/No)
T100-94	---	0.50	0.08	---	---	Yes
T100-95	99.44	0.270	0.027	2	0.054	Yes

“—” The registered participant did not submit the results.

“---” Data or information was not provided.

N/A: nominated but not registered laboratory

* Lab T100-75 reported the range of value.

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Table V: Participants' results, reported uncertainties, and laboratory accreditation status for lead

Lab Code	Mass fraction of lead (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor, <i>k</i> (95% level of confidence)	Expanded uncertainty (mg/kg)	Laboratory accreditation (Yes/No)
T100-01	0.302	0.006	2	0.013	Yes
T100-02	0.308	0.0203	2	0.0406	Yes
T100-03	0.38	0.015	2	0.030	Yes
T100-04	0.31	0.032	2.0	0.064	Yes
T100-05	0.25	0.045	2	0.09	Yes
T100-06	ND	---	---	---	No
T100-07	0.295	0.03	2	0.05	Yes
T100-08	0.3944	0.0016	2	0.0033	No
T100-09	0.27	0.013	2	0.025	Yes
T100-10	0.286	0.100	---	---	Yes
T100-11	0.276	0.013	4.303	0.058	Yes
T100-12	0.29	0.015	2	0.03	Yes
T100-13	0.239	0.1415	2	0.2830	Yes
T100-14	0.192	0.048	2	0.096	Yes
T100-15	0.254	107000	2	214000	Yes
T100-16	0.333	0.0047	2	0.0093	Yes
T100-17	0.293	0.0130	2	0.0260	Yes
T100-18	0.341	0.024	2	0.048	Yes
T100-19	0.25	0.0002	2	0.127	Yes
T100-20	0.51	0.014	2	0.0295	Yes
T100-21	0.412	0.044	2	0.088	Yes
T100-22	0.260	0.0019	2	0.004	No
T100-23	0.294	0.014	2.57	0.007	No
T100-24	0.26	0.038	2	0.077	Yes
T100-25	0.3	0.02	2.01	0.04	Yes
T100-26	0.33	---	---	0.01	Yes
T100-27	0.267	0.037	2	0.075	Yes
T100-28	0.2841	0.027	2	0.054	Yes
T100-29	0.567	0.065	2	0.130	Yes
T100-30	0.303	0.0424	2	0.0848	No
T100-31	0.277	0.005	2	0.010	No
T100-32	N/A	N/A	N/A	N/A	Yes
T100-33	0.288	0.03	2	0.06	Yes



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Lab Code	Mass fraction of lead (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor, <i>k</i> (95% level of confidence)	Expanded uncertainty (mg/kg)	Laboratory accreditation (Yes/No)
T100-34	0.257	0.046	2	0.092	Yes
T100-35	0.249	0.0063	2	0.0105	Yes
T100-36	0.261	0.017	2	0.035	Yes
T100-37	0.226	0.038	2	0.077	Yes
T100-38	0.300	0.016	2	0.032	Yes
T100-39	0.290	0.0309	2	0.0618	Yes
T100-40	0.111	±0.005	---	---	No
T100-41	—	—	—	—	Yes
T100-42	0.375853	0.035345	2	0.070690	Yes
T100-43	0.29	-	-	-	Yes
T100-44	0.20	0.03	2	0.06	Yes
T100-45	N/A	N/A	N/A	N/A	Yes
T100-46	0.296	0.004	2	0.01	Yes
T100-47	0.7725	0.0123	2	0.0246	Yes
T100-48	0.3380	0.0498	2	0.0996	Yes
T100-49	0.304	0.046	2	0.091	Yes
T100-50	0.27	0.01	2.8	0.028	Yes
T100-51	0.280	0.051	2	0.103	Yes
T100-52	0.26	---	---	---	Yes
T100-53	0.280	0.03	2	0.06	Yes
T100-54	N/A	N/A	N/A	N/A	Yes
T100-55	0.275	0.037	2	0.074	Yes
T100-56	0.35	0.05	2	0.10	Yes
T100-57	0.286	0.02	2	0.04	Yes
T100-58	0.165	0.04	0.05	0.09	Yes
T100-59	0.248	0.037	2	0.074	Yes
T100-60	N/A	N/A	N/A	N/A	Yes
T100-61	0.24	---	---	---	Yes
T100-62	0.248	0.007	2	0.014	Yes
T100-63	0.365	0.052	2	0.104	Yes
T100-64	—	—	—	—	Yes
T100-65	0.250	0.0034	2	0.068	No
T100-66	0.31	0.022	2	0.044	Yes
T100-67	0.242	0.0088	2	0.013	Yes



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Lab Code	Mass fraction of lead (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor, <i>k</i> (95% level of confidence)	Expanded uncertainty (mg/kg)	Laboratory accreditation (Yes/No)
T100-68	0.37	±0.03	2.0	0.06	Yes
T100-69	0.273	---	---	0.028	Yes
T100-70	0.23	0.015	2	0.030	Yes
T100-71	—	—	—	—	Yes
T100-72	1.12	---	2	---	No
T100-73	0.529	0.0318	2	0.0636	Yes
T100-74	0.24	0.12	2	0.2	Yes
T100-75	<9.1	---	---	---	Yes
T100-76	0.54	0.006	2	0.012	Yes
T100-77	0.259	0.011	2	0.022	Yes
T100-78	0.42	0.05	2	0.10	Yes
T100-79	0.159	0.00828	2.0	0.0166	Yes
T100-80	0.25	0.006	2	0.012	Yes
T100-81	0.58	0.0154	2	0.0307	Yes
T100-82	0.24	0.012	2	0.024	Under
T100-83	0.27	0.01	2	0.02	Yes
T100-84	0.285	0.019	2	0.038	Yes
T100-85	0.280	0.021	2	0.042	Yes
T100-86	0.2	0.04	2	0.07	Yes
T100-87	0.280	0.056	2	0.112	Yes
T100-88	0.1	---	---	---	No
T100-89	0.30	0.033	1.96	0.06	Yes
T100-90	0.260	---	---	---	Yes
T100-91	0.255	0.0383	2	0.0765	Yes
T100-92	0.239	---	---	0.011	Yes
T100-93	0.222	0.00733	2	0.01465	Yes
T100-94	0.40	0.06	---	---	Yes
T100-95	0.410	0.041	2	0.082	Yes

“—” The registered participant did not submit the results.

“---” Data or information was not provided.

N/A: nominated but not registered laboratory

* Lab T100-75 reported the range of value



Table VI: Summary of participants' digestion technique, medium and matrix separation for cadmium analysis

Lab Code	Digestion technique	Digestion medium	Matrix separation?
T100-01	Wet digestion	HNO ₃ /HCl	Yes
T100-02	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-03	Microwave-assisted	5mLHNO ₃ + 2mLH ₂ O ₂	No
T100-04	Microwave-assisted	HNO ₃	No
T100-05	Wet digestion	HNO ₃ /H ₂ O ₂	No
T100-06	Wet digestion	HNO ₃ /HCl/HClO ₄	Yes
T100-07	closed vessel / hot block	HNO ₃ /HCl	No
T100-08	Microwave-assisted	HNO ₃	Yes
T100-09	Wet digestion	HNO ₃ /HClO ₄	Yes
T100-10	Microwave-assisted	HNO ₃ /HCl/H ₂ O ₂	No
T100-11	Microwave-assisted	HNO ₃ /HCl	No
T100-12	Microwave-assisted	HNO ₃ /HCl	No
T100-13	Microwave-assisted	HNO ₃ / H ₂ O ₂ (Suprapur)	No
T100-14	Microwave-assisted	HNO ₃ /H ₂ O ₂	Yes
T100-15	Microwave-assisted	HNO ₃	No
T100-16	Microwave-assisted	HNO ₃ /HCl/H ₂ O ₂	---
T100-17	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-18	Dry ashing	HNO ₃	No
T100-19	Microwave-assisted	HNO ₃	---
T100-20	Wet digestion / Dry ashing	HNO ₃ /HCl	No
T100-21	Dry ashing	HNO ₃ /HCl/H ₂ O ₂	Yes
T100-22	Wet digestion	HNO ₃ /H ₂ SO ₄ /HClO ₄	No
T100-23	Microwave-assisted digestion ; close vessel digestion	HNO ₃	No
T100-24	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-25	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-26	Wet digestion	HNO ₃	---
T100-27	Microwave-assisted	HNO ₃ /HCl	No
T100-28	Microwave-assisted	HNO ₃	No
T100-29	Microwave-assisted	HNO ₃	Yes
T100-30	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-31	Microwave-assisted	HNO ₃	No
T100-32	N/A	N/A	N/A
T100-33	Microwave-assisted	HNO ₃	Yes



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Lab Code	Digestion technique	Digestion medium	Matrix separation?
T100-34	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-35	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-36	Microwave-assisted	HNO ₃	Yes
T100-37	Dry ashing	---	No
T100-38	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-39	Microwave-assisted	HNO ₃ /H ₂ O ₂	Yes
T100-40	Dry ashing	HCl	No
T100-41	—	—	—
T100-42	Wet digestion	HNO ₃ /H ₂ SO ₄ /H ₂ O ₂	Yes
T100-43	Wet digestion	HNO ₃ /H ₂ O ₂	No
T100-44	Microwave-assisted	HNO ₃	No
T100-45	N/A	N/A	N/A
T100-46	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-47	Dry ashing	HCl	No
T100-48	Microwave-assisted digestion (FOCT 31671-2012)	HNO ₃	Yes
T100-49	Dry ashing	---	No
T100-50	Microwave-assisted	HNO ₃	No
T100-51	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-52	Wet digestion	HNO ₃ /H ₂ O ₂	No
T100-53	Microwave-assisted	HNO ₃	No
T100-54	N/A	N/A	N/A
T100-55	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-56	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-57	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-58	Microwave-assisted	HNO ₃	Yes
T100-59	Microwave-assisted	HNO ₃	No
T100-60	N/A	N/A	N/A
T100-61	Microwave-assisted	HNO ₃	No
T100-62	Microwave-assisted	HNO ₃	No
T100-63	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-64	—	—	—
T100-65	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-66	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-67	Wet Digestion	HNO ₃ and HF	No
T100-68	Dry ashing	HNO ₃ /HCl	No



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Lab Code	Digestion technique	Digestion medium	Matrix separation?
T100-69	Dry ashing	HNO ₃	No
T100-70	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-71	—	—	—
T100-72	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-73	Microwave-assisted	HNO ₃	No
T100-74	Microwave-assisted	HNO ₃	No
T100-75	Wet digestion via ASTM D3335-08	HNO ₃	No
T100-76	Dry ashing	HNO ₃	No
T100-77	Dry ashing	HNO ₃	No
T100-78	Microwave-assisted	HNO ₃	No
T100-79	Dry ashing	HCl	No
T100-80	Wet digestion	HNO ₃ /H ₂ O ₂	No
T100-81	Dry ashing	HNO ₃	No
T100-82	Microwave-assisted	HNO ₃	Yes
T100-83	Microwave-assisted	HNO ₃	No
T100-84	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-85	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-86	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-87	Microwave-assisted	HNO ₃	No
T100-88	Dry ashing	HNO ₃	Yes
T100-89	Microwave-assisted	HNO ₃	Yes
T100-90	Microwave-assisted	HNO ₃	No
T100-91	Microwave-assisted	HNO ₃	Yes
T100-92	Microwave-assisted	HNO ₃ /H ₂ O ₂ /HCl	No
T100-93	Microwave-assisted	HNO ₃	Yes
T100-94	Microwave-assisted	HNO ₃ /H ₂ O ₂	Yes
T100-95	Dry ashing	HNO ₃	Yes



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Table VII: Summary of participants' digestion technique, medium and matrix separation for lead analysis

Lab Code	Digestion technique	Digestion medium	Matrix separation?
T100-01	Wet digestion	HNO ₃ /HCl	Yes
T100-02	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-03	Microwave-assisted	5mLHNO ₃ + 2mLH ₂ O ₂	No
T100-04	Microwave-assisted	HNO ₃	No
T100-05	Wet digestion	HNO ₃ /H ₂ O ₂	No
T100-06	Wet digestion	HNO ₃ /HCl/HClO ₄	Yes
T100-07	closed vessel / hot block	HNO ₃ /HCl	No
T100-08	Microwave-assisted	HNO ₃	No
T100-09	Wet digestion	HNO ₃ /HClO ₄	Yes
T100-10	Dry ashing	HNO ₃ /HCl	No
T100-11	Microwave-assisted	HNO ₃ /HCl	No
T100-12	Microwave-assisted	HNO ₃ /HCl	No
T100-13	Microwave-assisted	HNO ₃ / H ₂ O ₂ (Suprapur)	-
T100-14	Microwave-assisted	HNO ₃ /H ₂ O ₂	Yes
T100-15	Microwave-assisted	HNO ₃	No
T100-16	Microwave-assisted	HNO ₃ /HCl/H ₂ O ₂	-
T100-17	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-18	Dry ashing	HNO ₃	No
T100-19	Microwave-assisted	HNO ₃	-
T100-20	Wet digestion / Dry ashing	HNO ₃ /HCl	No
T100-21	Dry ashing	HNO ₃ /HCl/H ₂ O ₂	Yes
T100-22	Wet digestion	HNO ₃ /H ₂ SO ₄ /HClO ₄	No
T100-23	Microwave-assisted digestion ; close vessel digestion	HNO ₃	No
T100-24	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-25	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-26	Wet digestion	HNO ₃	-
T100-27	Microwave-assisted	HNO ₃ /HCl	No
T100-28	Microwave-assisted	HNO ₃	No
T100-29	Microwave-assisted	HNO ₃	Yes
T100-30	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-31	Microwave-assisted	HNO ₃	No
T100-32	N/A	N/A	N/A
T100-33	Microwave-assisted	HNO ₃	Yes



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Lab Code	Digestion technique	Digestion medium	Matrix separation?
T100-34	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-35	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-36	Microwave-assisted	HNO ₃	Yes
T100-37	Dry ashing	----	No
T100-38	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-39	Microwave-assisted	HNO ₃ /H ₂ O ₂	Yes
T100-40	Dry ashing	HCl	No
T100-41	—	—	—
T100-42	Wet digestion	HNO ₃ /H ₂ SO ₄ /H ₂ O ₂	Yes
T100-43	Wet digestion	HNO ₃ /H ₂ O ₂	No
T100-44	Microwave-assisted	HNO ₃	No
T100-45	N/A	N/A	N/A
T100-46	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-47	Dry ashing	HCl	No
T100-48	Microwave-assisted	HNO ₃	Yes
T100-49	Dry ashing	N/A	No
T100-50	Microwave-assisted	HNO ₃	No
T100-51	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-52	Wet digestion	HNO ₃ /H ₂ O ₂	No
T100-53	Microwave-assisted	HNO ₃	No
T100-54	N/A	N/A	N/A
T100-55	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-56	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-57	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-58	Microwave-assisted	HNO ₃	Yes
T100-59	Microwave-assisted	HNO ₃	No
T100-60	N/A	N/A	N/A
T100-61	Microwave-assisted	HNO ₃	No
T100-62	Microwave-assisted	HNO ₃	No
T100-63	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-64	—	—	—
T100-65	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-66	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-67	Wet Digestion	HNO ₃ and HF	No
T100-68	Dry ashing	HNO ₃ /HCl	No



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Lab Code	Digestion technique	Digestion medium	Matrix separation?
T100-69	Dry ashing	HNO ₃	No
T100-70	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-71	—	—	—
T100-72	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-73	Microwave-assisted	HNO ₃	No
T100-74	Microwave-assisted	HNO ₃	No
T100-75	Wet digestion	HNO ₃	No
T100-76	Dry ashing	HNO ₃	No
T100-77	Dry ashing	HNO ₃	No
T100-78	Microwave-assisted	HNO ₃	No
T100-79	Dry ashing	HCl	No
T100-80	Wet digestion	HNO ₃ /H ₂ O ₂	No
T100-81	Dry ashing	HNO ₃	No
T100-82	Microwave-assisted	HNO ₃	Yes
T100-83	Microwave-assisted	HNO ₃	No
T100-84	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-85	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-86	Microwave-assisted	HNO ₃ /H ₂ O ₂	No
T100-87	Microwave-assisted	HNO ₃	No
T100-88	Dry ashing	HNO ₃	Yes
T100-89	Microwave-assisted	HNO ₃	No
T100-90	Microwave-assisted	HNO ₃	No
T100-91	Microwave-assisted	HNO ₃	Yes
T100-92	Microwave-assisted	HNO ₃ /H ₂ O ₂ /HCl	No
T100-93	Microwave-assisted	HNO ₃	Yes
T100-94	Microwave-assisted	HNO ₃ /H ₂ O ₂	Yes
T100-95	Dry ashing	HNO ₃	Yes



Table VIII: Summary of participants' information on the use of internal standard, correction for recovery, instrumental method and method validation status for cadmium analysis

Lab Code	Use of internal standard?	Correction for recovery?	Instrumental method	Method validation?
T100-01	Yes	Yes	ICP-MS	Yes
T100-02	Yes	No	ICP-MS	Yes
T100-03	No	Yes	Graphite AAS	Yes
T100-04	No	No	ICP-MS	No
T100-05	No	No	Inversion voltammetry	Yes
T100-06	No	No	Flame AAS	No
T100-07	Yes	No	ICP-MS	Yes
T100-08	No	Yes	ICP-OES	No
T100-09	Yes	No	ICP-AES	Yes
T100-10	No	No	Graphite AAS	No
T100-11	Yes	No	ICP-MS	Yes
T100-12	Yes	No	ICP-MS	Yes
T100-13	No	No	ICP-OES	Yes
T100-14	No	No	Graphite AAS	Yes
T100-15	No	No	ICP-MS	Yes
T100-16	Yes	No	ICP-MS	Yes
T100-17	Yes	No	ICP-MS	No
T100-18	No	No	Graphite AAS	No
T100-19	----	----	ICP-MS	Yes
T100-20	Yes	No	Graphite AAS	Yes
T100-21	Yes	No	Graphite AAS	Yes
T100-22	Yes	No	ICP-MS	Yes
T100-23	Yes	Yes	ICP-MS	Yes
T100-24	No	No	Graphite AAS	Yes
T100-25	Yes	No	ICP-MS	Yes
T100-26	Yes	No	ICP-MS	Yes
T100-27	Yes	No	ICP-MS	Yes
T100-28	No	No	Graphite AAS	Yes
T100-29	No	Yes	Graphite AAS	Yes
T100-30	Yes	No	ICP-MS	Yes
T100-31	Yes	No	ICP-MS	Yes
T100-32	N/A	N/A	N/A	N/A



Lab Code	Use of internal standard?	Correction for recovery?	Instrumental method	Method validation?
T100-33	Yes	No	ICP-MS	No
T100-34	No	No	Graphite AAS	Yes
T100-35	No	No	Graphite AAS	Yes
T100-36	Yes	No	ICP-MS	Yes
T100-37	No	No	ICP-AES	Yes
T100-38	Yes	No	ICP-MS	pending
T100-39	No	Yes	Graphite AAS	No
T100-40	No	Yes	Graphite AAS	No
T100-41	—	—	—	—
T100-42	Yes	No	ICP-OES	Yes
T100-43	Yes	No	ICP-MS	No
T100-44	No	No	ICP-AES	Yes
T100-45	N/A	N/A	N/A	N/A
T100-46	No	No	Graphite AAS	Yes
T100-47	No	No	Graphite AAS	-
T100-48	No	No	Flame AAS	No
T100-49	No	No	Polarograph Metrohm	Yes
T100-50	No	Yes	ICP-AES	Yes
T100-51	Yes	No	ICP-MS	No
T100-52	No	Yes	Graphite AAS	No
T100-53	Yes	No	ICP-AES	Yes
T100-54	N/A	N/A	N/A	N/A
T100-55	Yes	No	ICP-MS	Yes
T100-56	No	No	Graphite AAS	Yes
T100-57	No	No	ICP-MS	Yes
T100-58	Yes	Yes	ICP-MS	Yes
T100-59	Yes	No	ICP-MS	Yes
T100-60	N/A	N/A	N/A	N/A
T100-61	No	No	ICP-MS	Yes
T100-62	Yes	No	ICP-MS	Yes
T100-63	No	No	Graphite AAS	Yes
T100-64	—	—	—	—
T100-65	No	No	Graphite AAS	No
T100-66	Yes	Yes	ICP-MS	Yes



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Lab Code	Use of internal standard?	Correction for recovery?	Instrumental method	Method validation?
T100-67	Yes	No	ICP-MS	Yes
T100-68	No	No	ICP-AES	No
T100-69	Yes	No	ICP-MS	No
T100-70	Yes	No	ICP-AES	Yes
T100-71	—	—	—	—
T100-72	---	No	Flame AAS	Yes
T100-73	No	No	Graphite AAS	Yes
T100-74	No	No	ICP-MS	Yes
T100-75	Yes	No	Flame AAS	Yes
T100-76	Yes	No	Graphite AAS	No
T100-77	No	No	Flame AAS	Yes
T100-78	Yes	Yes	Flame AAS and Graphite AAS	Yes
T100-79	No	No	Flame AAS	No
T100-80	No	No	Graphite AAS	Yes
T100-81	No	Yes	Graphite AAS	Yes
T100-82	No	Yes	ICP-OES	Yes
T100-83	No	No	ICP-OES	No
T100-84	Yes	No	ICP-MS	Yes
T100-85	No	No	Graphite AAS	Yes
T100-86	No	No	ICP-MS	Yes
T100-87	Yes	No	ICP-MS	Yes
T100-88	No	No	Flame AAS	In progress
T100-89	No	Yes	ICP-MS	Yes
T100-90	Yes	No	ICP-MS	Yes
T100-91	Yes	No	Graphite AAS	Yes
T100-92	Yes	Yes	ICP-MS	Yes
T100-93	No	No	Graphite AAS	Yes
T100-94	Yes	No	Graphite AAS	Yes
T100-95	Yes	No	Flame AAS	Yes



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Table IX: Summary of participants' information on the use of internal standard, correction for recovery, instrumental method and method validation status for lead analysis

Lab Code	Use of internal standard?	Correction for recovery?	Instrumental method	Method validation?
T100-01	Yes	Yes	ICP-MS	Yes
T100-02	Yes	No	ICP-MS	Yes
T100-03	No	Yes	Graphite AAS	Yes
T100-04	No	No	ICP-MS	No
T100-05	No	No	Inversion voltammetry	Yes
T100-06	No	No	Flame AAS	No
T100-07	Yes	No	ICP-MS	Yes
T100-08	No	Yes	ICP-OES	No
T100-09	Yes	No	ICP-AES	Yes
T100-10	No	No	Graphite AAS	No
T100-11	Yes	No	ICP-MS	Yes
T100-12	Yes	No	ICP-MS	Yes
T100-13	No	No	ICP-OES	Yes
T100-14	No	No	Graphite AAS	Yes
T100-15	No	No	ICP-MS	Yes
T100-16	Yes	No	ICP-MS	Yes
T100-17	Yes	No	ICP-MS	No
T100-18	No	No	Graphite AAS	No
T100-19	----	----	ICP-MS	Yes
T100-20	Yes	No	Graphite AAS	Yes
T100-21	Yes	No	Graphite AAS	Yes
T100-22	Yes	No	ICP-MS	Yes
T100-23	Yes	Yes	ICP-MS	Yes
T100-24	No	No	Graphite AAS	Yes
T100-25	Yes	No	ICP-MS	Yes
T100-26	Yes	No	ICP-MS	Yes
T100-27	Yes	No	ICP-MS	Yes
T100-28	No	No	Graphite AAS	Yes
T100-29	No	Yes	Graphite AAS	Yes
T100-30	Yes	No	ICP-MS	Yes
T100-31	Yes	No	ICP-MS	Yes
T100-32	N/A	N/A	N/A	N/A



Lab Code	Use of internal standard?	Correction for recovery?	Instrumental method	Method validation?
T100-33	Yes	No	ICP-MS	No
T100-34	No	No	Graphite AAS	Yes
T100-35	No	No	Graphite AAS	Yes
T100-36	Yes	No	ICP-MS	Yes
T100-37	No	No	ICP-AES	Yes
T100-38	Yes	No	ICP-MS	pending
T100-39	No	Yes	Graphite AAS	No
T100-40	No	Yes	Graphite AAS	No
T100-41	—	—	—	—
T100-42	Yes	No	ICP-OES	Yes
T100-43	Yes	No	ICP-MS	No
T100-44	No	No	ICP-AES	Yes
T100-45	N/A	N/A	N/A	N/A
T100-46	No	No	Graphite AAS	Yes
T100-47	No	No	Graphite AAS	-
T100-48	No	No	Flame AAS	No
T100-49	No	No	Polarograph Metrohm	Yes
T100-50	No	Yes	ICP-AES	Yes
T100-51	Yes	No	ICP-MS	No
T100-52	No	Yes	Graphite AAS	No
T100-53	Yes	No	ICP-AES	Yes
T100-54	N/A	N/A	N/A	N/A
T100-55	Yes	No	ICP-MS	Yes
T100-56	No	No	Graphite AAS	Yes
T100-57	No	No	ICP-MS	Yes
T100-58	Yes	Yes	ICP-MS	Yes
T100-59	Yes	No	ICP-MS	Yes
T100-60	N/A	N/A	N/A	N/A
T100-61	No	No	ICP-MS	Yes
T100-62	Yes	No	ICP-MS	Yes
T100-63	No	No	Graphite AAS	Yes
T100-64	—	—	—	—
T100-65	No	No	Graphite AAS	No
T100-66	Yes	Yes	ICP-MS	Yes



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Lab Code	Use of internal standard?	Correction for recovery?	Instrumental method	Method validation?
T100-67	Yes	No	ICP-MS	Yes
T100-68	No	No	ICP-AES	No
T100-69	Yes	No	ICP-MS	No
T100-70	Yes	No	ICP-AES	Yes
T100-71	—	—	—	—
T100-72	---	No	Flame AAS	Yes
T100-73	No	No	Graphite AAS	Yes
T100-74	No	No	ICP-MS	Yes
T100-75	Yes	No	Flame AAS	Yes
T100-76	Yes	No	Graphite AAS	No
T100-77	No	No	Flame AAS	Yes
T100-78	Yes	Yes	Flame AAS and Graphite AAS	Yes
T100-79	No	No	Flame AAS	No
T100-80	No	No	Graphite AAS	Yes
T100-81	No	Yes	Graphite AAS	Yes
T100-82	No	Yes	ICP-OES	Yes
T100-83	No	No	ICP-OES	No
T100-84	Yes	No	ICP-MS	Yes
T100-85	No	No	Graphite AAS	Yes
T100-86	No	No	ICP-MS	Yes
T100-87	Yes	No	ICP-MS	Yes
T100-88	No	No	Flame AAS	In progress
T100-89	No	Yes	ICP-MS	Yes
T100-90	Yes	No	ICP-MS	Yes
T100-91	Yes	No	Graphite AAS	Yes
T100-92	Yes	Yes	ICP-MS	Yes
T100-93	No	No	Graphite AAS	Yes
T100-94	Yes	No	Graphite AAS	Yes
T100-95	Yes	No	Flame AAS	Yes



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Figure I: Plot of z-scores of participants on cadmium results

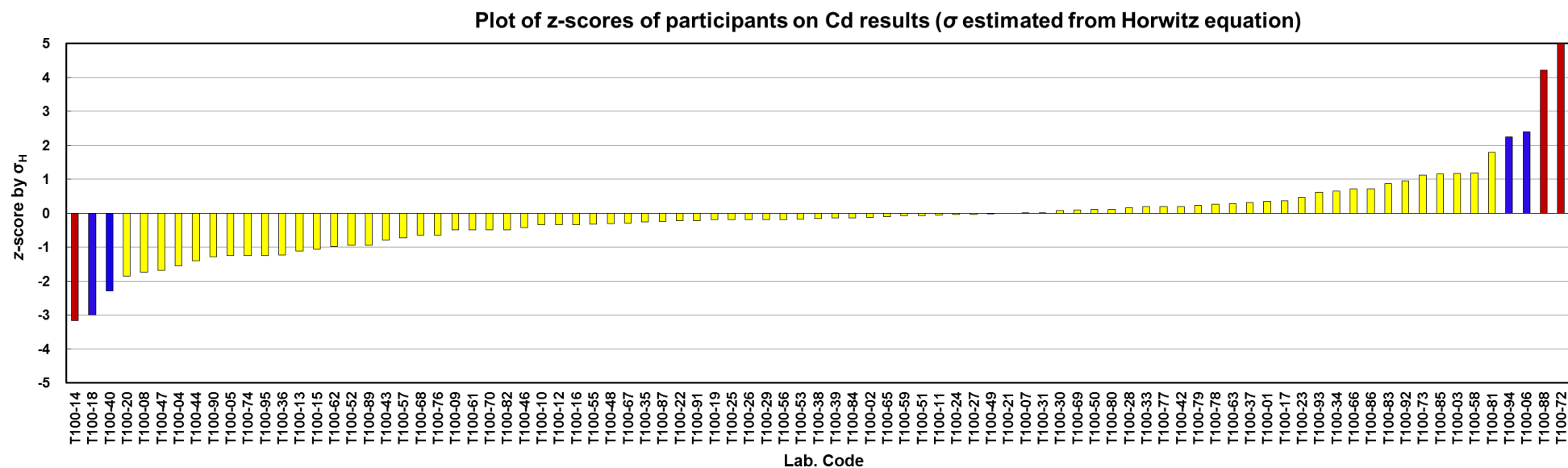
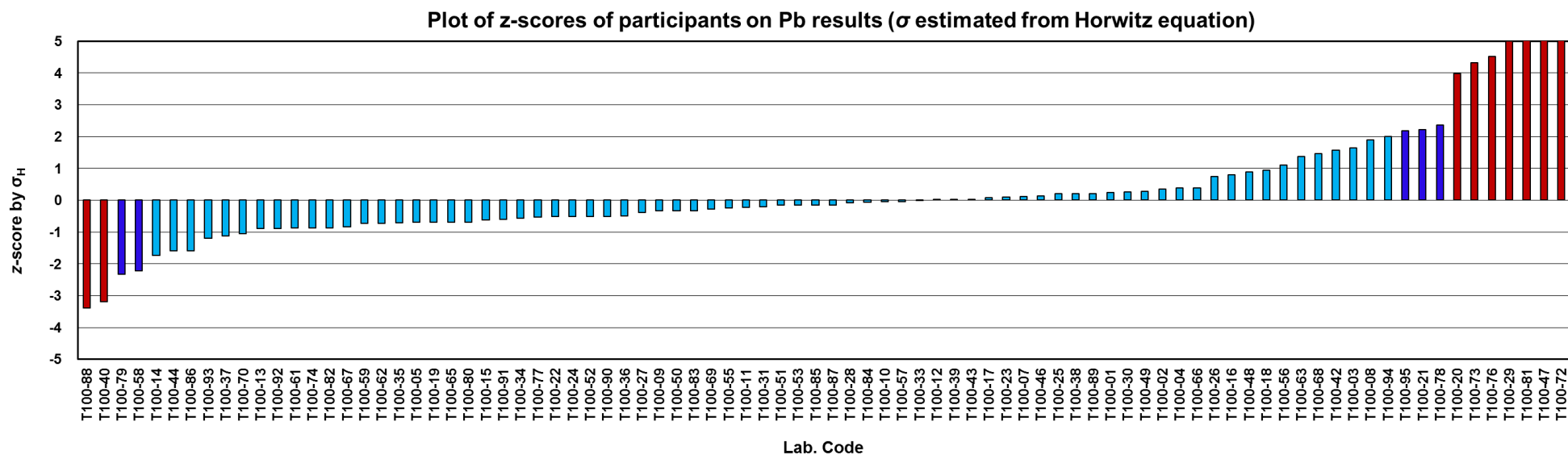




Figure II: Plot of z-scores of participants on lead results

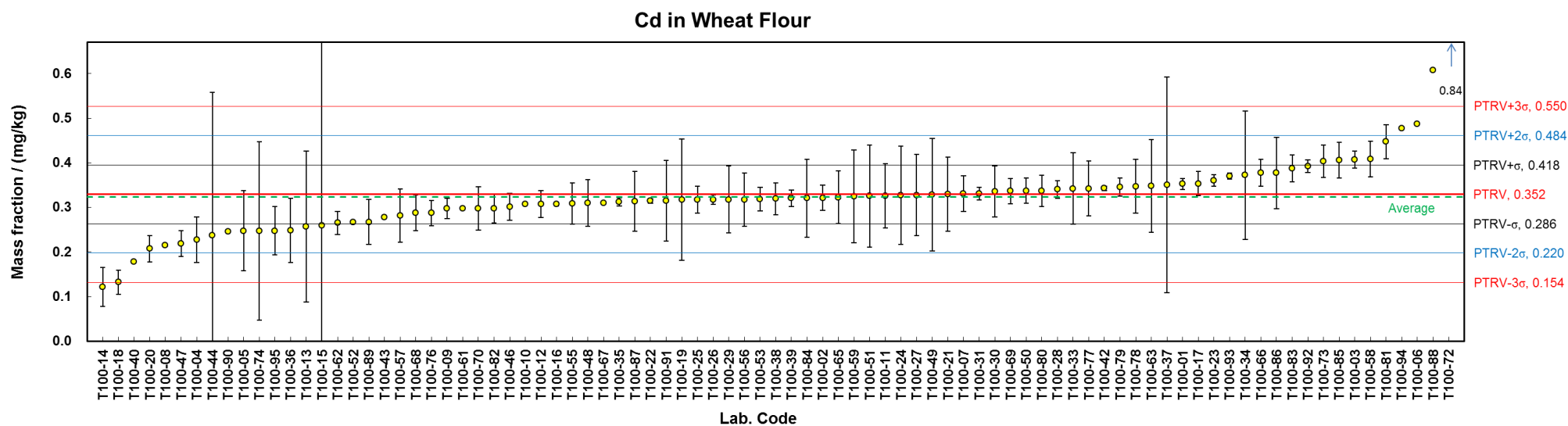




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Figure III: PTRV for cadmium and participants results





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Figure IV: PTRV for lead and participants results

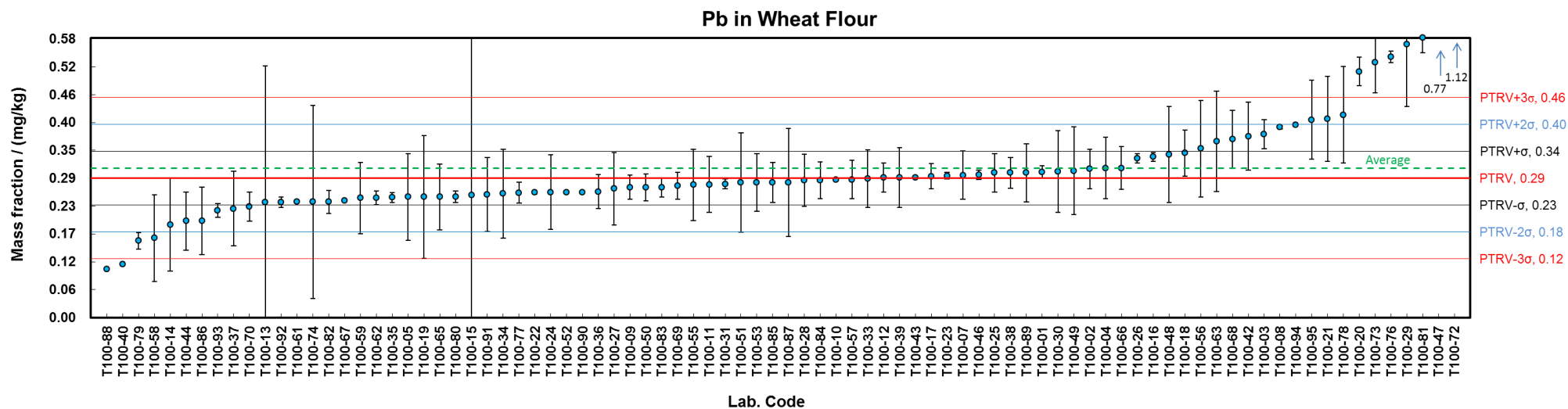




Figure V: Youden plot showing distribution of participants' results

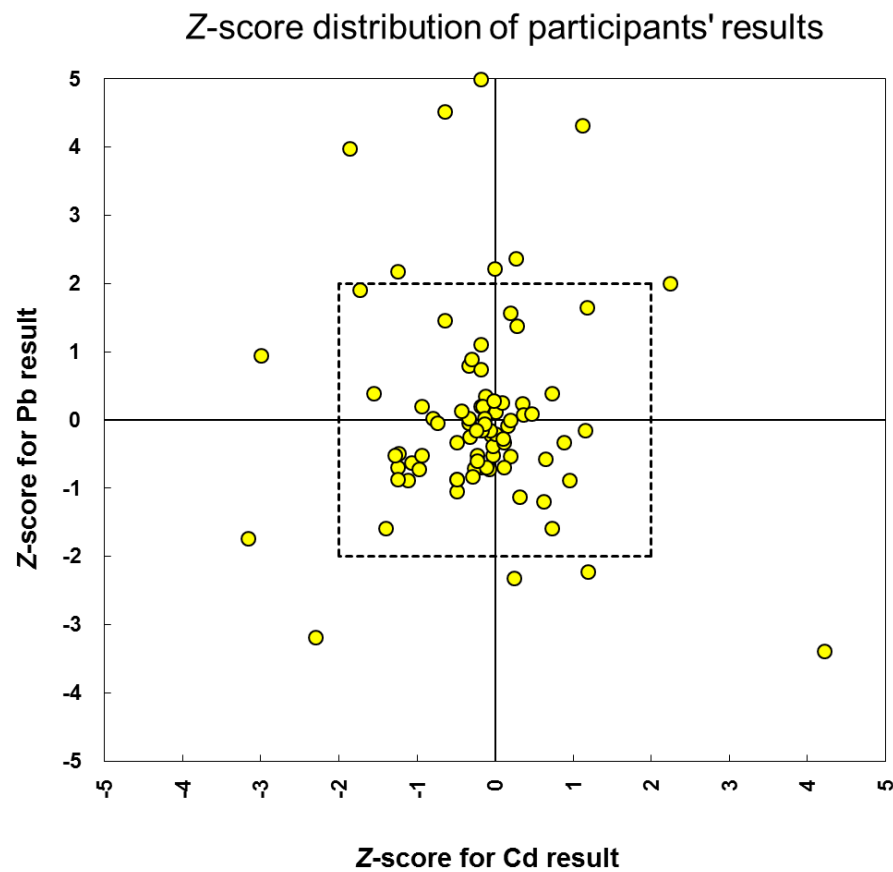




Figure VI: Youden plot showing distribution of participants' results with respect to accreditation for the test

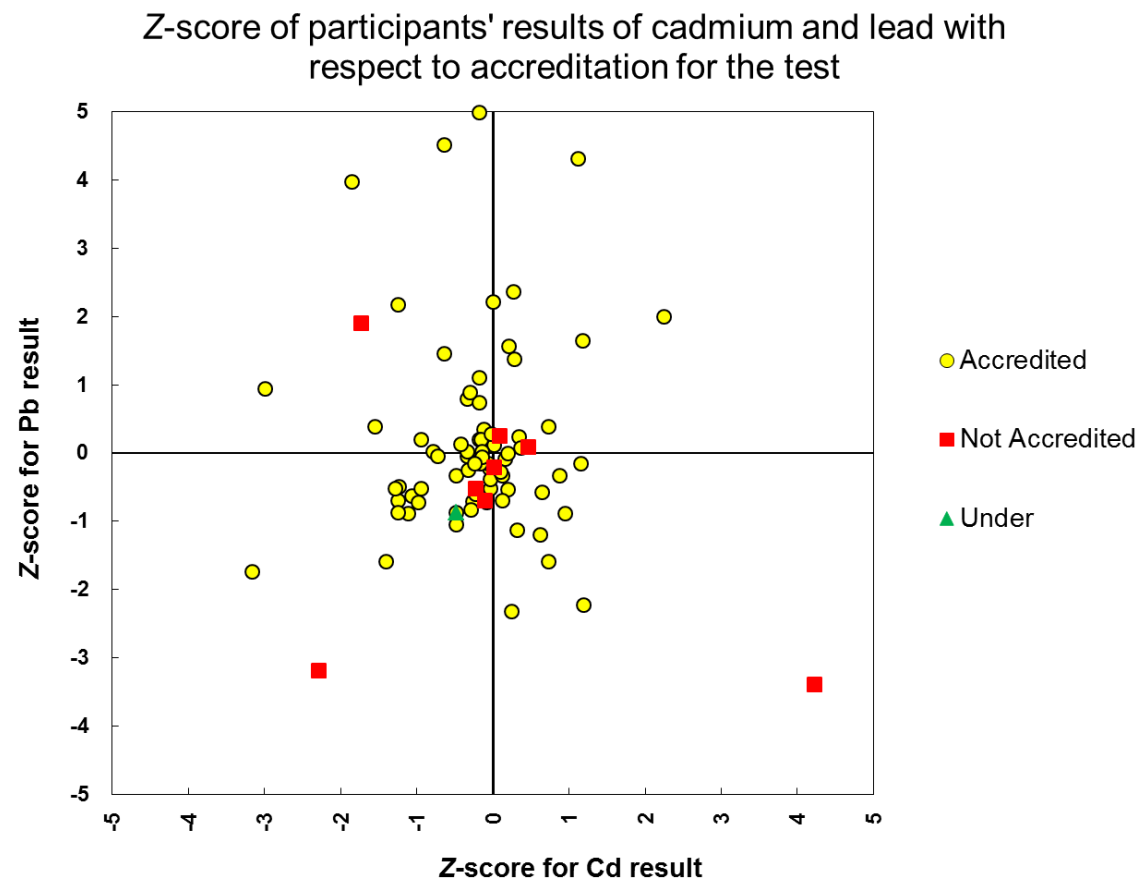
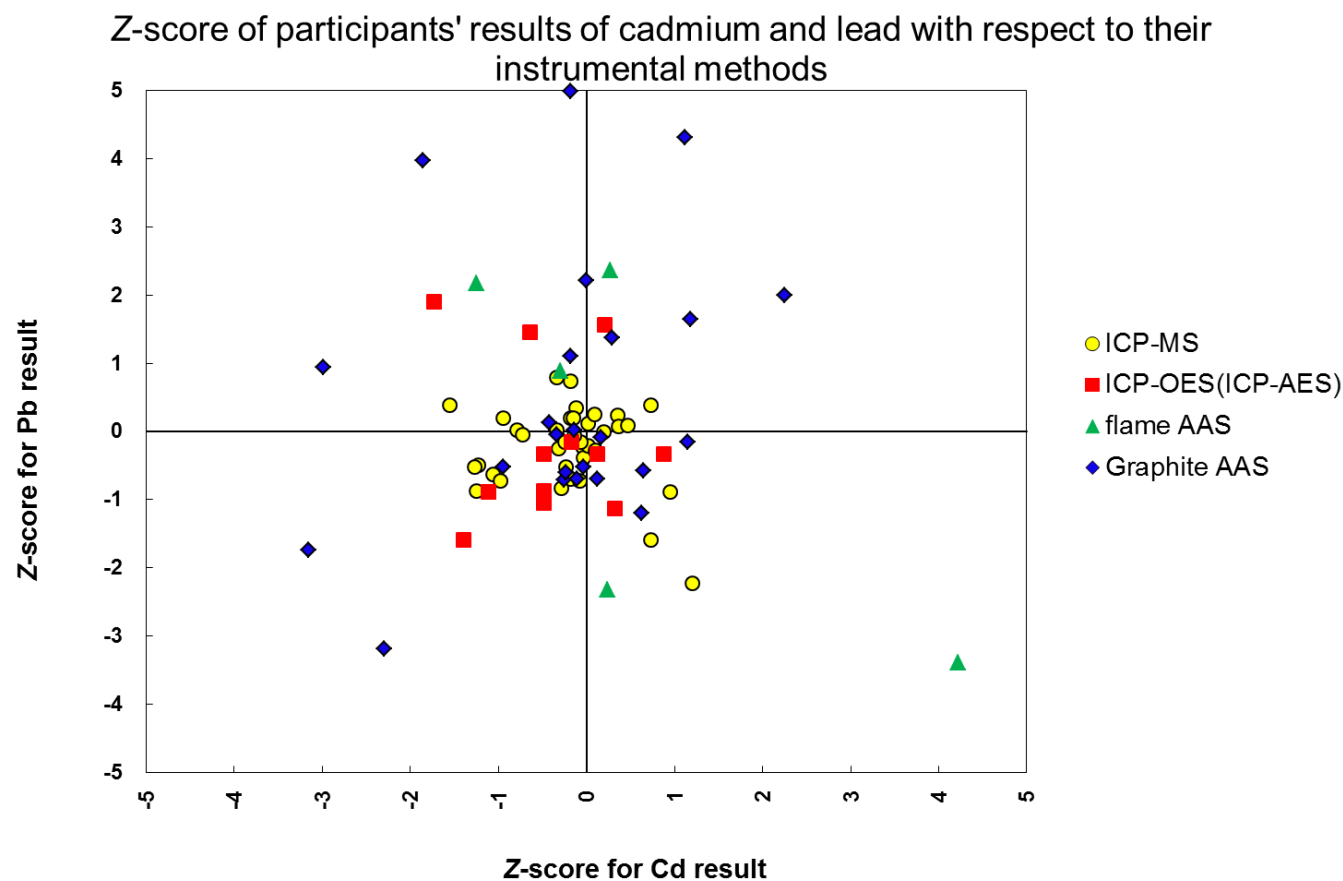




Figure VII: Youden plot showing distribution of participants' results with respect to their instrumental methods





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Figure VIII: Results from all participants according to participants' instrumental methods

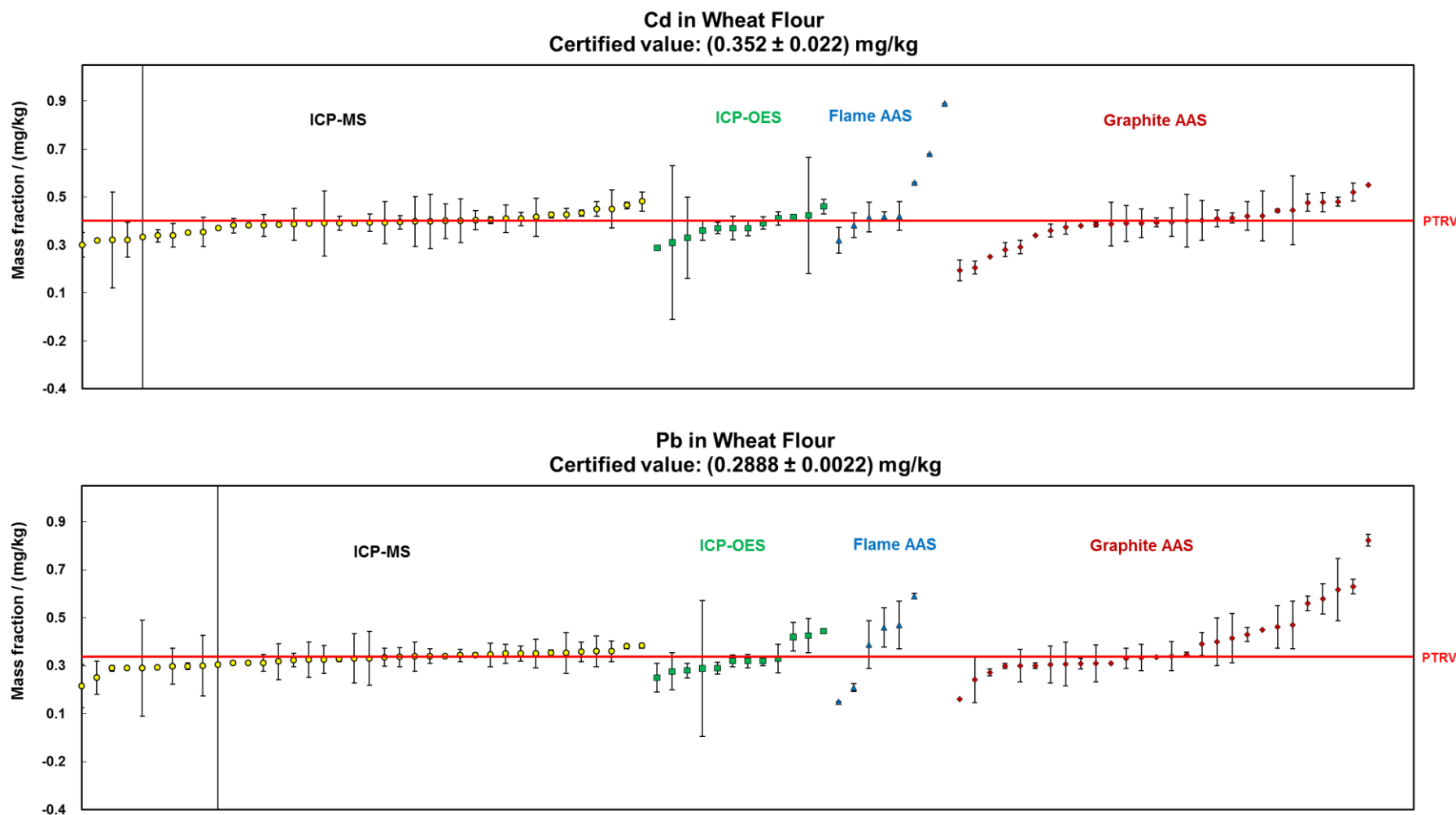
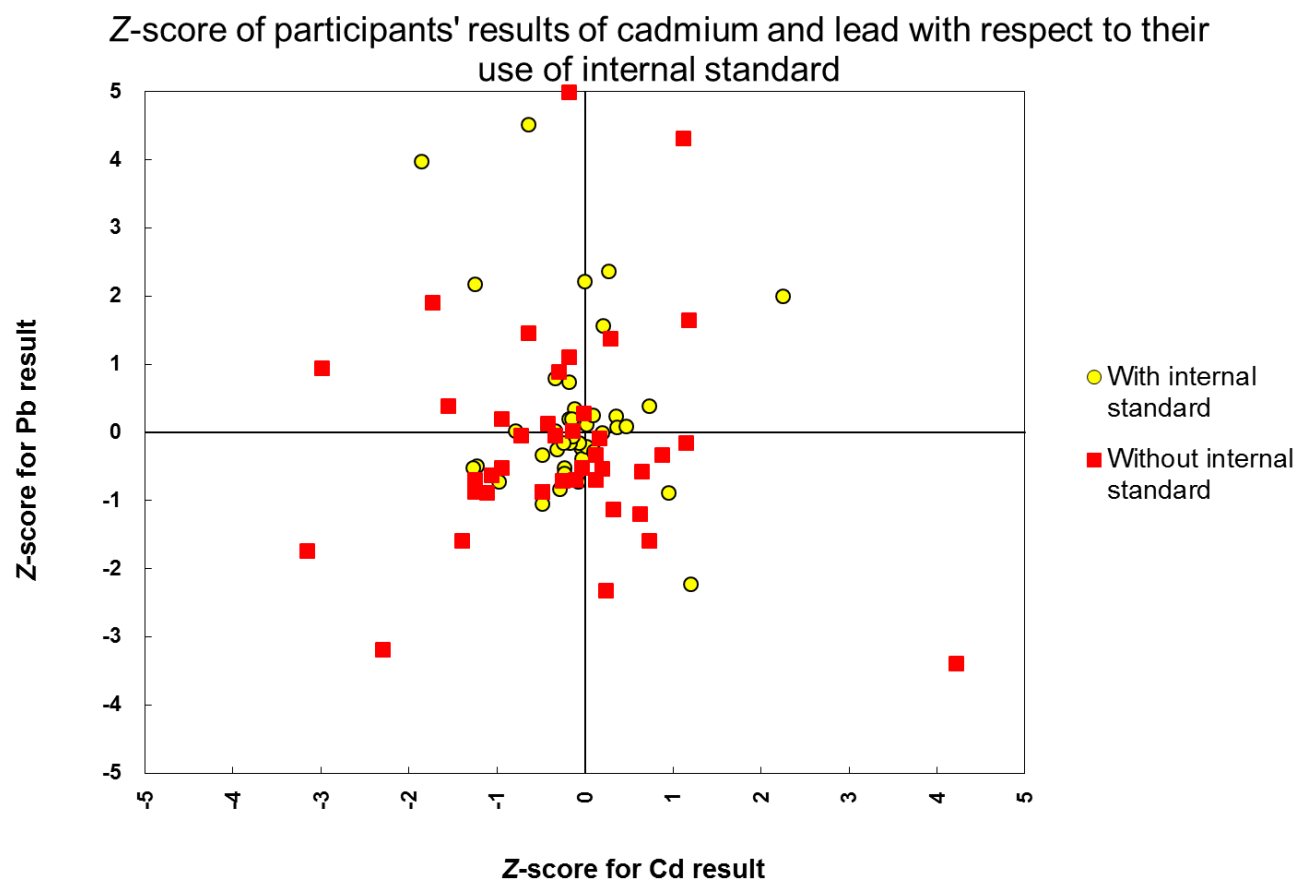




Figure IX: Youden plot showing distribution of participants' results with respect to the use of internal standard





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Figure X: Results from all participants according to the use of internal standard

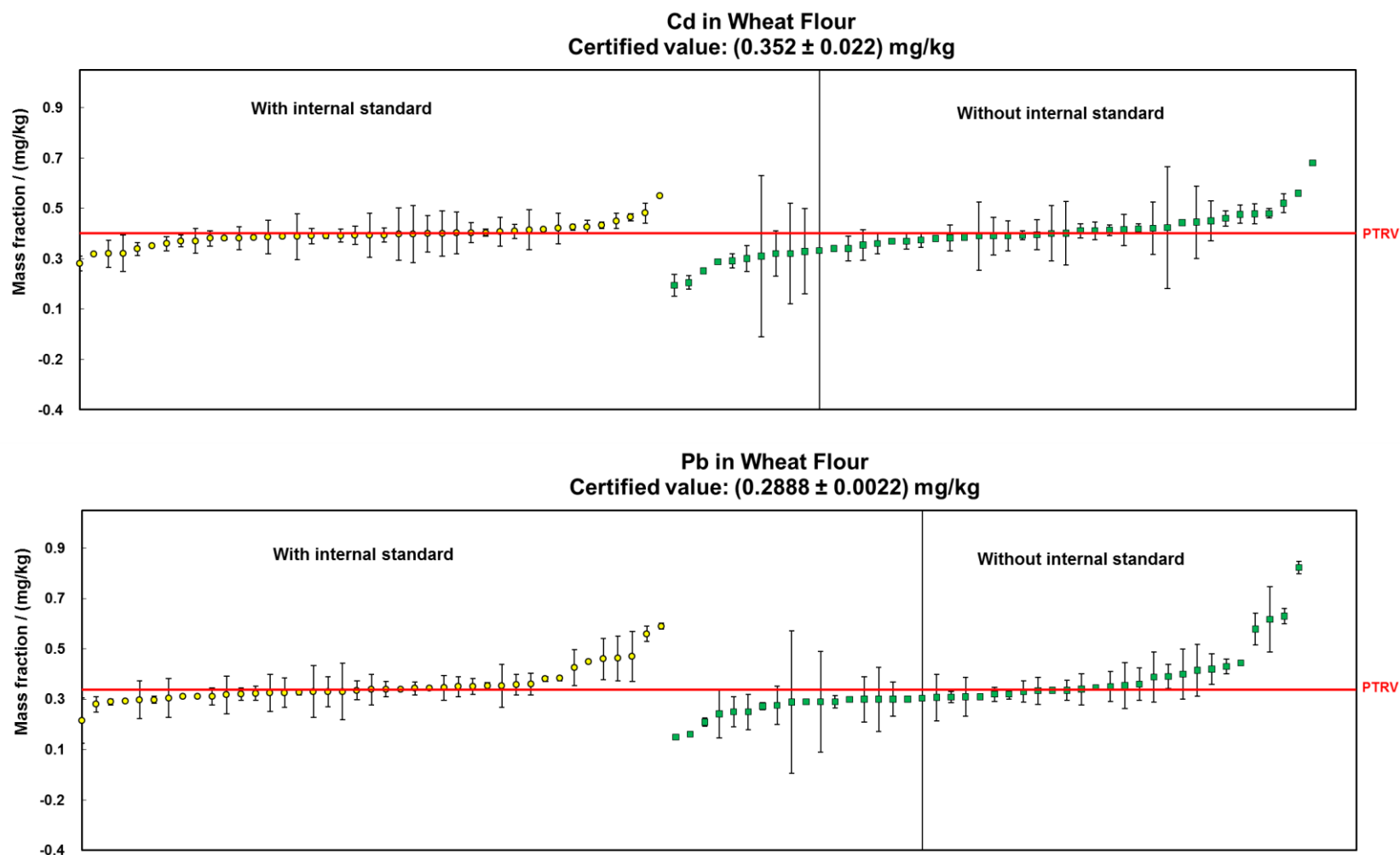
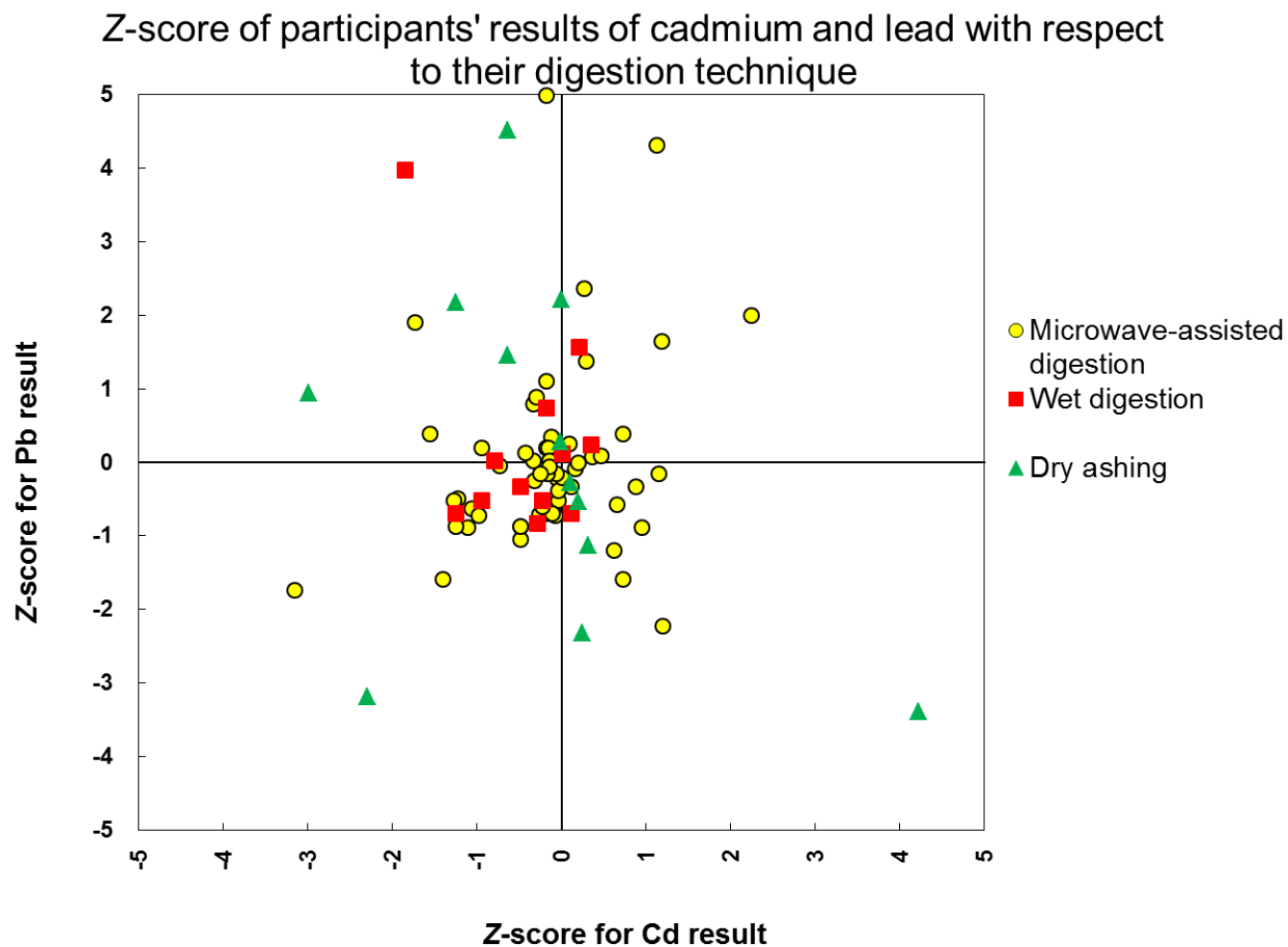




Figure XI: Youden plot showing distribution of participants' results with respect to their digestion technique





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Figure XII: Results from all participants according to their digestion technique

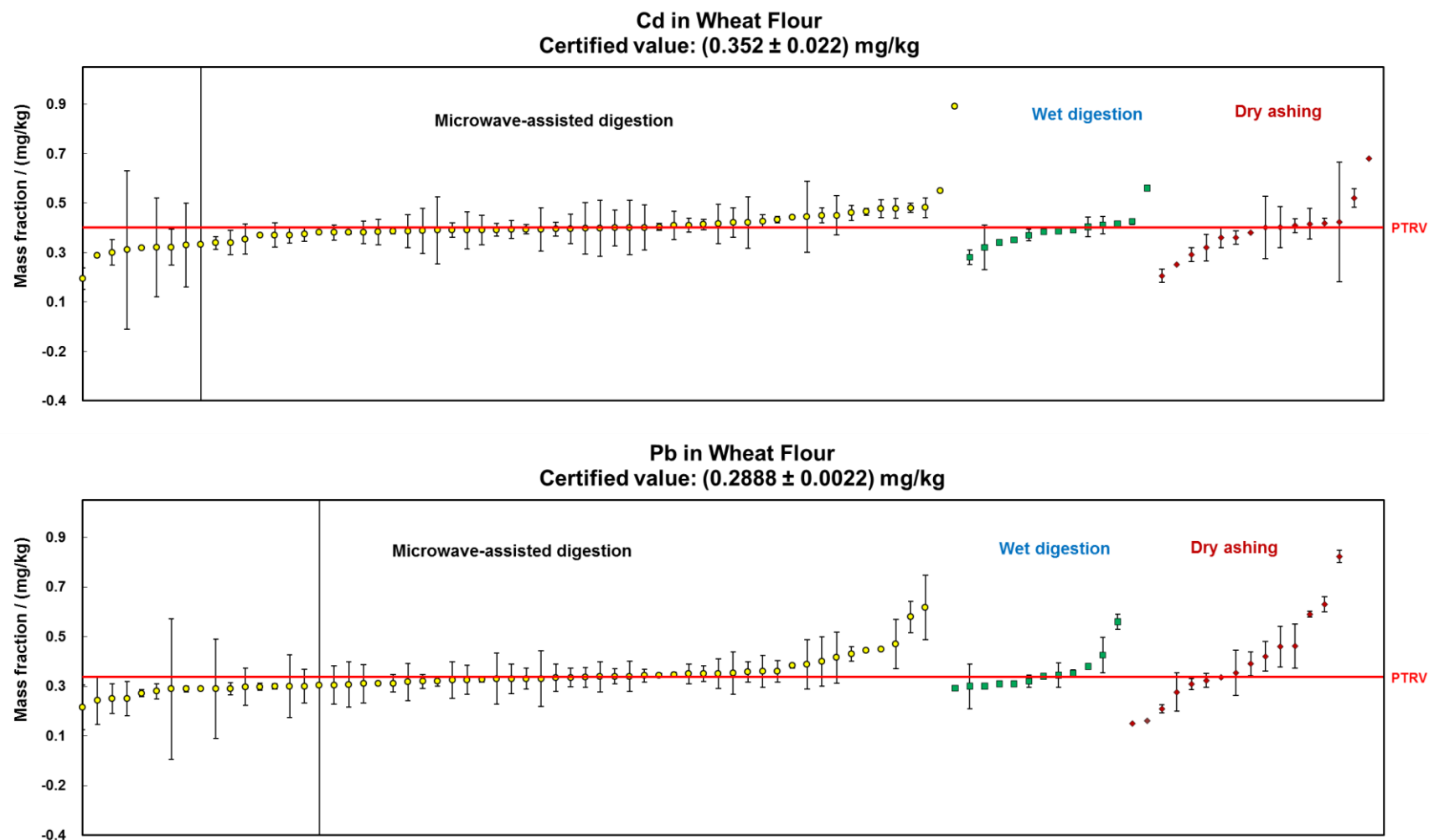
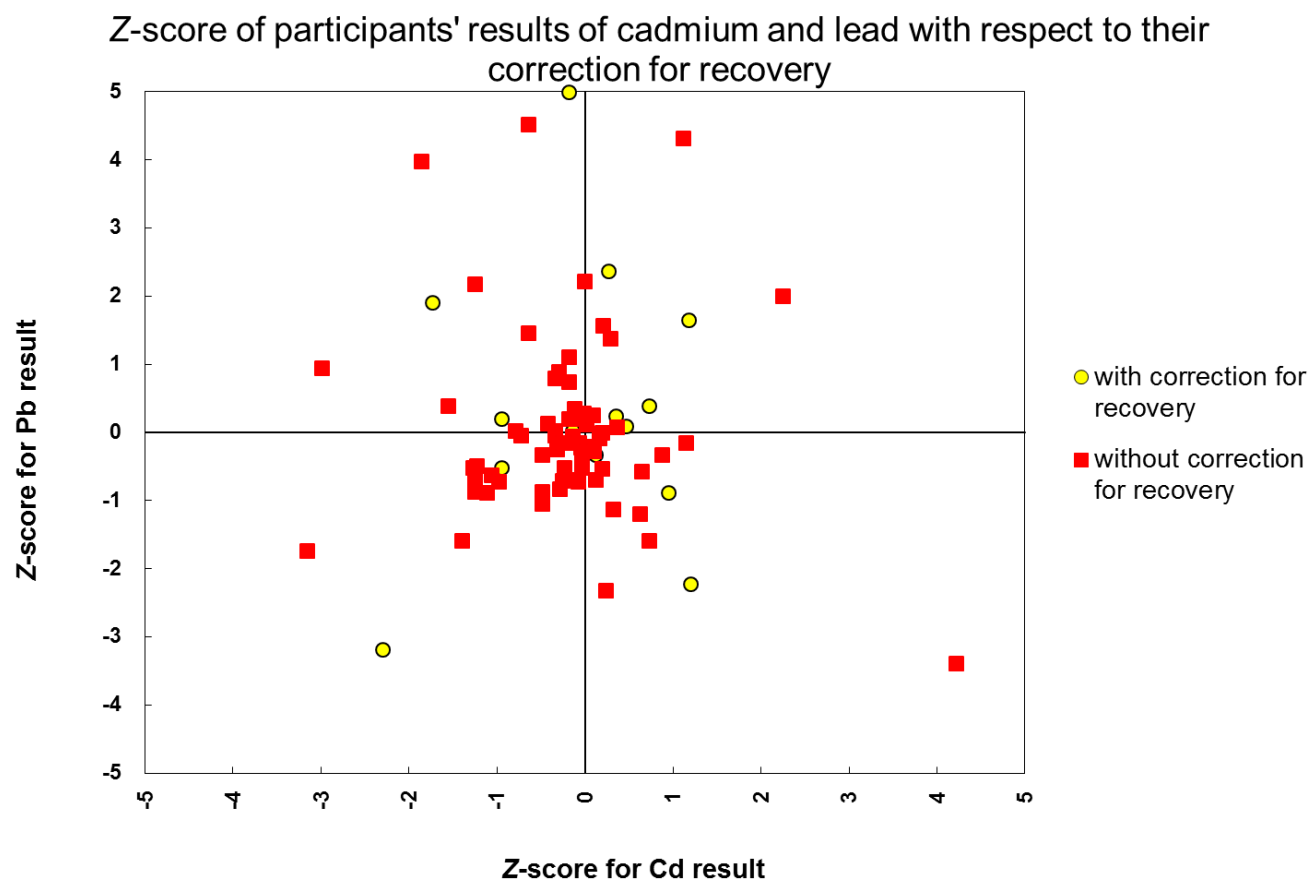




Figure XIII: Youden plot showing distribution of participants' results with respect to the application of recovery correction





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Figure XIV: Results from all participants according to the application of recovery correction

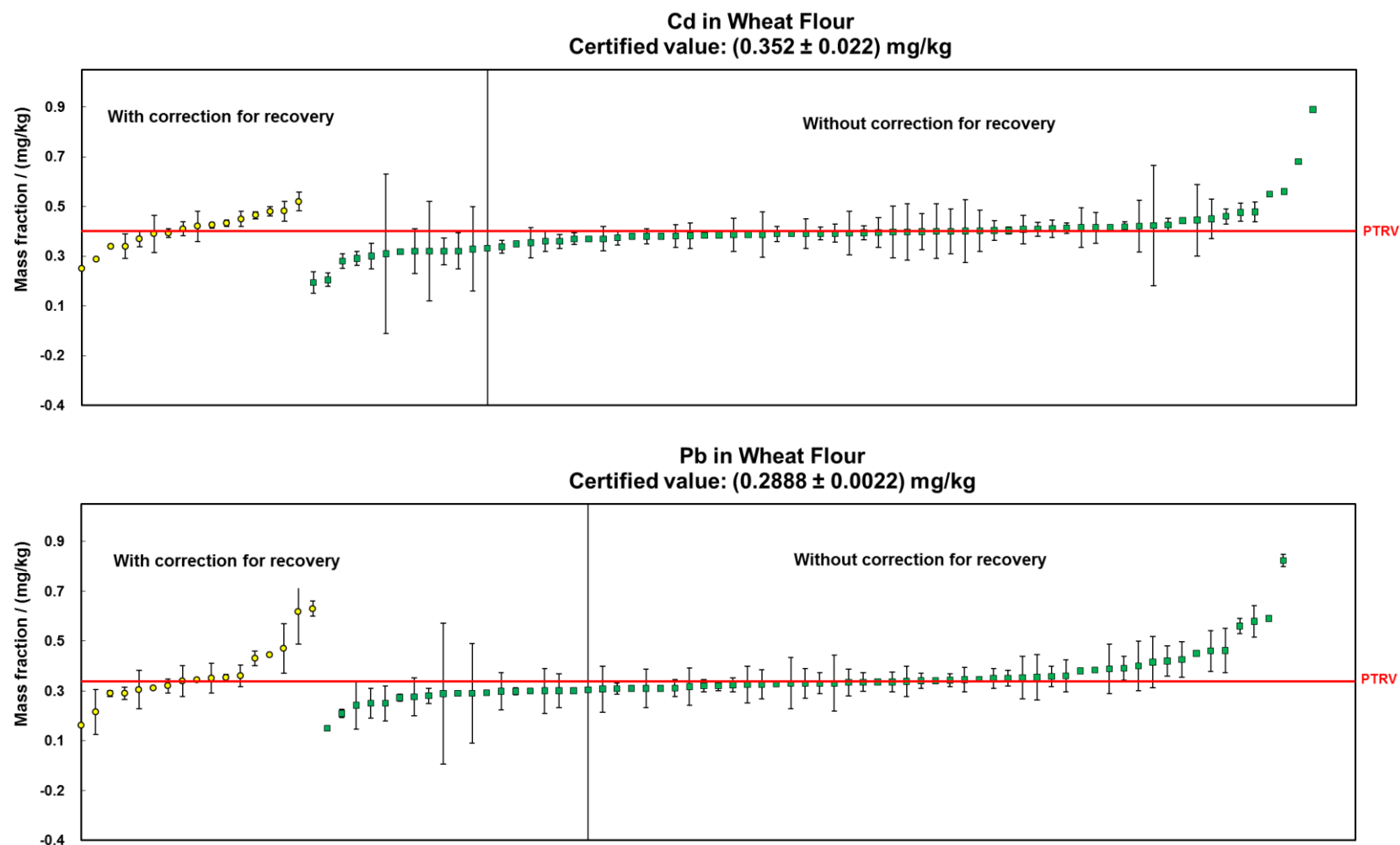
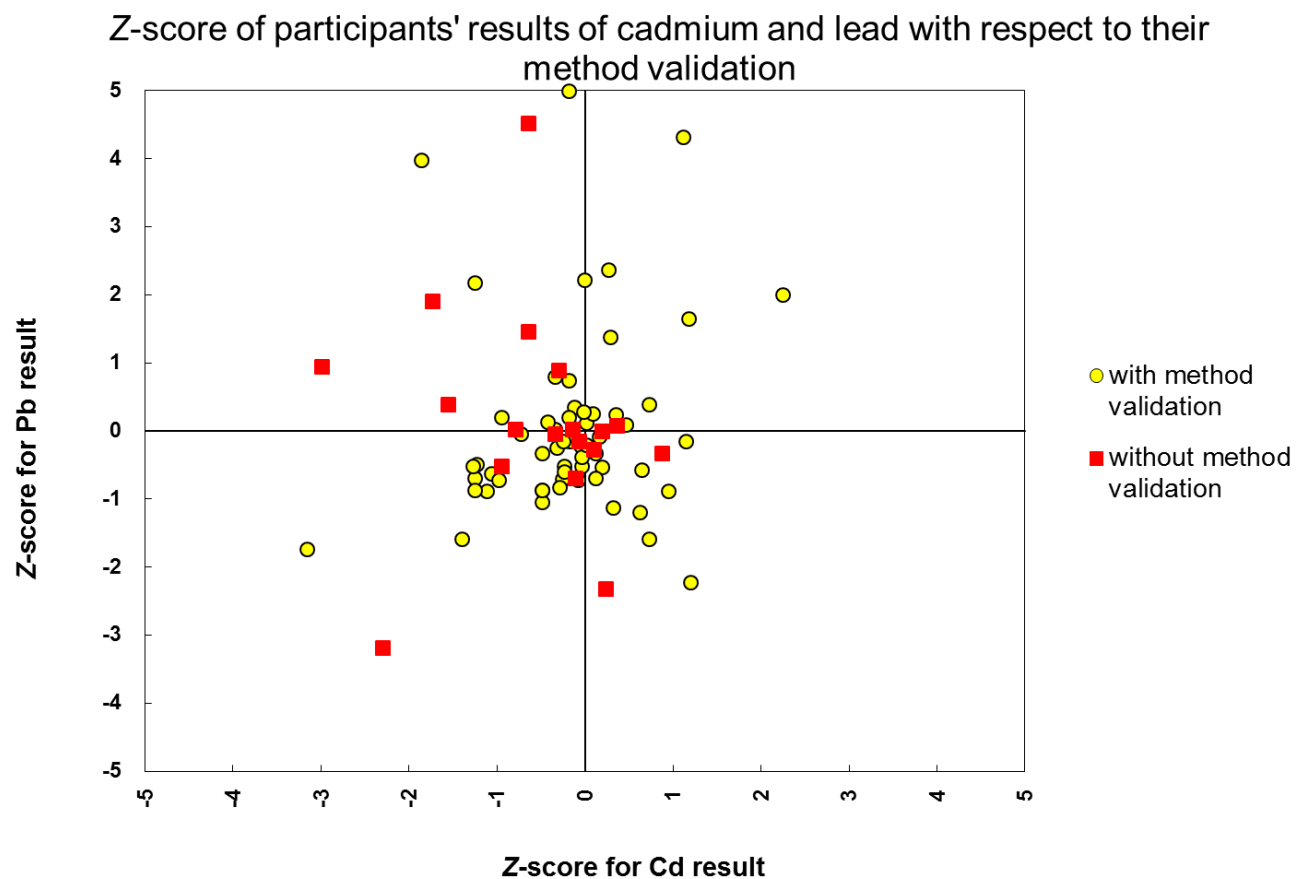




Figure XV: Youden plot showing distribution of participants' results with respect to their method validation

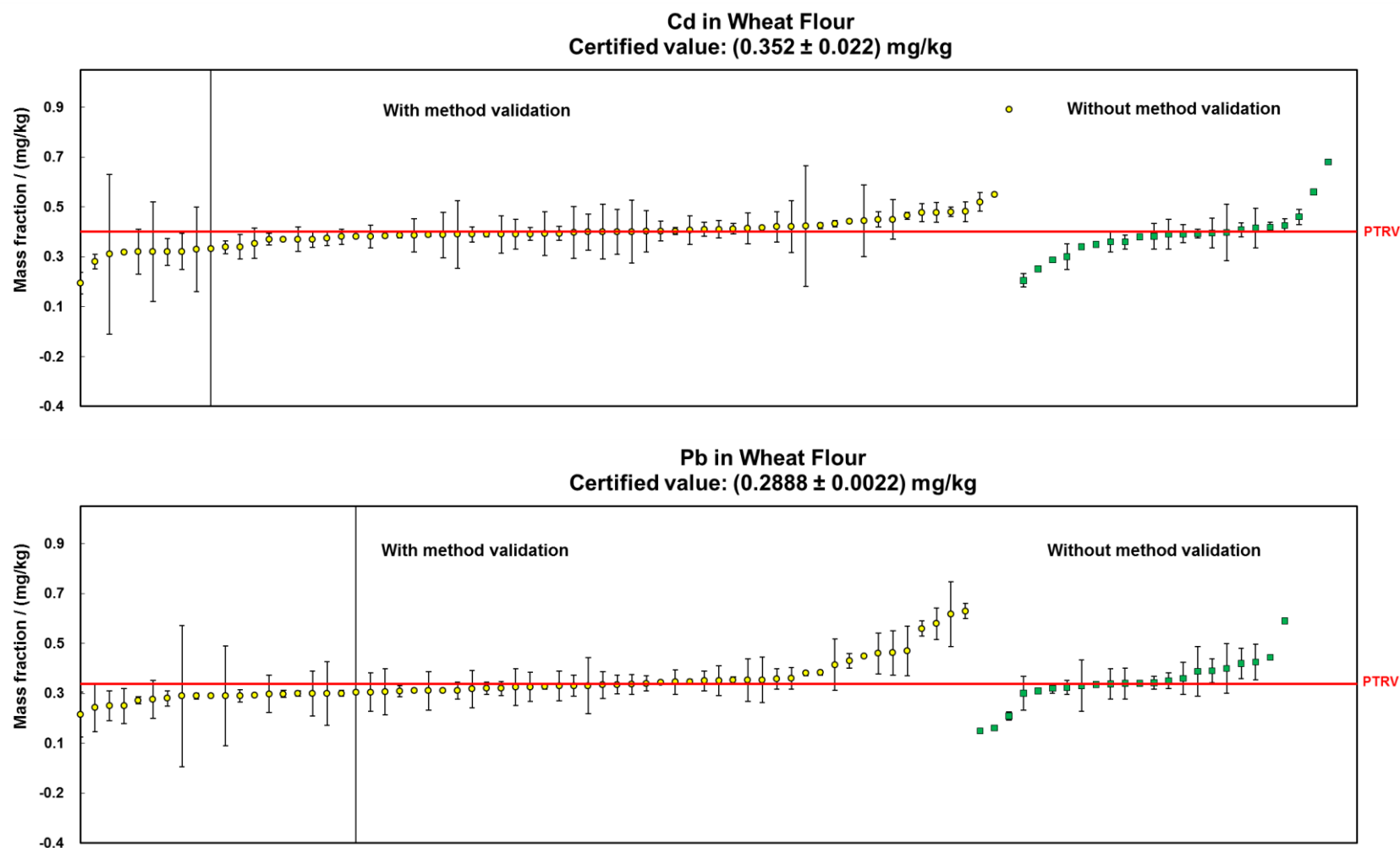




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Figure XVI: Results from all participants according to their method validation





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APPENDIX I: Instruction to Participants

Instructions for Participants

1. Analysis of the proficiency test sample

(1) Sample information

- Participating laboratories will be provided with **ONE** bottle containing about **20 g** of wheat flour. The analyte contents in each bottle should be considered equivalent after dry-mass correction within the level of the between-sample homogeneity. The date of dispatching of samples will be informed to participants.
- Participants are required to confirm the receipt of the samples (including the serial number of the sample, date of receipt, any damages found in the sample package) by e-mail (aplacpt.inorg@kriss.re.kr) using the sample receipt form attached in Annex A. If any damage on the sample is observed on arrival, please contact us immediately.**
- Analytes and their approximate mass fractions are given as follows.

Element	Mass fraction (expected range of values)
Cadmium (Cd)	0.05 mg/kg -1 mg/kg
Lead (Pb)	0.05 mg/kg -1 mg/kg

- Homogeneity: ID-ICP/MS analysis of one subsample taken from each of 11 or 12 bottles was carried out. The relative standard deviations of the results obtained from 12 and 11 bottles were 0.29 % and 0.23 % for Cd and Pb, respectively, which are satisfactory for the present proficiency testing.

(2) Sample storage

- The proficiency test sample should be kept sealed in its original bottle and stored under room temperature conditions.
- Opening of the sample bottle should be carefully planned to avoid contamination and deterioration of the sample.
- For safety considerations, the proficiency test sample should be handled with care to prevent inhaling the sample powder and getting into eyes. In the case of accidental exposure to sample, wash the exposed areas with plenty of water and consult physicians when necessary.
- For this proficiency testing programme, it is not required to return remaining sample.

(3) Guidelines for sample preparation and dry-mass correction



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- Sample preparation for analysis:
 - Sample handling in a laboratory with well-controlled humidity and temperature is recommended. Any kind of contamination should be avoided.
 - Sampling for the analysis should be carried out at the same time as sampling for the dry-mass correction.
 - Digest the wheat flour material completely, if the method used requires digestion.
 - The proficiency test sample should be mixed thoroughly before taking an aliquot of sample from a bottle.
 - The analysis should be conducted with a recommended sample size of at least 0.5 g.
 - Participants are recommended to perform at least triplicate measurements and report the mean and associated measurement uncertainty for each analyte as specified in the Result Report. Participants should estimate the measurement uncertainty using their own practice.
- Dry-mass correction:
 - Participants should also carry out the dry mass correction.
 - Sampling for the dry mass correction should be carried out at the same time as the sampling for analysis.
 - At least three separate portions from a sample bottle (with a recommended sample size of about 0.5 g for each portion) should be taken as dry mass correction samples and placed over P_2O_5 in a desiccator at room temperature for 7 days (168 hours).
 - Calculate the moisture content from the mass change observed in the three aliquots and use it for dry-mass correction.

(4) Measurement method

- Participants are expected to use the test method of their choice, which should be consistent with their routine procedures. If the laboratory is accredited, it is recommended to use the method listed in the scope of its accreditation.



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2. Reporting and submission of results

Participants should complete the Result Report (Annex B). The manners of reporting test results are as follows:

- For each analyte, the mean value of at least three independent measurements and its associated expanded uncertainty with 95 % level of confidence should be reported on a dry mass basis;
- Report the mass fractions of analytes in mg/kg for cadmium and lead; and
- Participants should provide information about the method of analysis.

Participants should be aware that any submitted results are considered final and accordingly such results and units should be thoroughly checked before submission. Participants should submit the Result Report electronically to the coordinator of the proficiency testing programme (E-mail: aplacpt.inorg@kriss.re.kr) before the deadline, **4 September 2015**. Results submitted after the deadline will not be accepted. Participants are reminded that the ability to report results in the specified unit and within the given time scale are part of the proficiency test.

The proficiency testing programme is conducted in the belief that participants will perform the analysis and report results with scientific rigour. Collusion and falsification of results are clearly against the spirit of the proficiency testing programme.

3. Contact

Participants may wish to contact the coordinator of the proficiency testing programme for any enquires (E-mail: aplacpt.inorg@kriss.re.kr):

Dr. Sook Heun Kim
Senior Research Scientist
Center for Inorganic Analysis
Division of Metrology for Quality of Life
Korea Research Institute of Standards and Science (KRIS)
267 Gajeong-Ro, Yuseong-Gu, Daejeon 305-340, Republic of Korea
Tel: +82-42-868-5756
Fax: +82-42-868-5801



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APPENDIX II: Sample Receipt Form

Sample Receipt Form (Participating Laboratories)

Institute/
Laboratory:

Postal address:

Contact person:

Title

Given name

Surname

E-mail:

Date of Receipt:

Confirmation of Package Content

Please choose the state of the sample: ☐ intact

☐ broken

☐ others: _____

Please write the serial numbers of the sample bottle: _____

Please complete this form and return it to Dr. Sook Heun Kim, KRISS (E-mail: aplacpt.inorg@kriss.re.kr) by email immediately after receipt of the sample.



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APPENDIX III: Result Report Form

Result Report

Institute/
Laboratory: _____
Postal
address: _____
Contact
person: _____
Title Given name Surname

E-mail: _____

Laboratory accreditation: YES (based on ISO 17025/ ISO 9000 series/ GLP/ under the law of your economy(please specify)) /NO

If yes, please specify the name of accreditation body.

1. Analytical results

Analyte	Analytical result (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor k (95% level of confidence)	Expanded uncertainty (mg/kg)

Notes: (i) Report the analytical results and associated uncertainties in the unit mg/kg; (ii) Report the analytical results on a dry mass basis.



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1) Results from individual aliquot (dry-mass corrected)

number	Cd (mg/kg)	Pb (mg/kg)
1		
2		
3		
4		
Average		
Standard deviation		
Standard deviation of the mean		

Please insert more lines for more determinations.

2) Dry-mass Correction

Moisture content (% in mass): _____

2. Methods of analysis

1) Analyte: Cadmium

a) Sample size used for analysis : _____ g

b) Digestion technique: Microwave-assisted digestion / Wet digestion / Dry ashing
Others (please specify):

c) Digestion medium: HNO₃ / HCl / HF / H₂SO₄ / HClO₄ / H₂O₂ / Aqua regia
Others (please specify):

d) Matrix separation: YES / NO

e) Calibration method: External calibration / Internal calibration / Standard additions / Isotope dilution mass spectrometry

f) Source(s) of calibration standard(s): _____

g) Use of internal standard(s): YES (please specify): / NO

h) Analytical ICP-MS / ICP-AES / Flame AAS / Graphite AAS



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instrument(s): Others (please specify):

i) Correction for recovery: YES (please specify recovery (%)): / NO

j) Method validation: YES (please specify): / NO

k) Additional information: _____

2) Analyte: Lead

a) Sample size used for analysis: _____ g

b) Digestion technique: Microwave-assisted digestion / Wet digestion / Dry ashing
Others (please specify):

c) Digestion medium: HNO₃ / HCl / HF / H₂SO₄ / HClO₄ / H₂O₂ / Aqua regia
Others (please specify):

d) Matrix separation: YES / NO

e) Calibration method: External calibration / Internal calibration / Standard additions / Isotope dilution mass spectrometry

f) Source(s) of calibration standard(s): _____

g) Use of internal standard(s): YES (please specify): / NO

h) Analytical instrument(s): ICP-MS / ICP-AES / Flame AAS / Graphite AAS
Others (please specify):

i) Correction for recovery: YES (please specify recovery (%)): / NO



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j) Method validation: YES(please specify): / NO

k) Additional
information: _____

Print name of responsible person:

Date: ____ / ____ / ____.
dd mm yyyy