# Asia Pacific Metrology Programme (APMP) - Asia Pacific Laboratory Accreditation Cooperation (APLAC) Joint Proficiency Testing Programme <APLAC PT T105> Nutritional Elements (Iron and Zinc) 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 T105) 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 Science (KRISS), 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 nutritional elements, iron and zinc, in wheat flour with respect to the assignment of certified reference values.
2. The design of the proficiency testing (PT) 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. The 113 laboratories were nominated, 100 out of 113 laboratories from 45 economies enrolled in the program and 90 of them returned their results for iron and/or zinc. One laboratory (T105-081) for iron and three laboratories (T105-045, T105-065 and T105-081) for zinc reported the results as a range, not values. Two laboratories (T105-089 and T105-108) submitted only iron result. Participants were confidentially assigned with unique laboratory codes (T105-001 to T105-113) and the codes were used throughout the program.
4. The reference values certified by KRISS for zinc and iron in the test sample were used as the assigned values for evaluating the performance of participants of this PT. Isotope Dilution–Inductively Coupled Plasma/Mass Spectrometry (ID-ICP/MS) as a primary method was used for the certification. The standard deviations for performance assessment were calculated using the Horwitz Equation [3]. The *z*-scores and zeta-scores (ζ-scores) were used as numerical indicators to show participants' performance with respect to the assigned values in the program. The standard uncertainty from each participant was used to derive a zeta-score.

5. Performance of participants, in terms of *z*-scores, is summarized as follows:

| *z*-score | Number of Participants (Percentage) | |
|---|---|---|
| | Iron | Zinc |
| $\lvert z\rvert \le 2.0$ | 63 (71 %) | 60 (71 %) |
| $2.0 < \lvert z\rvert < 3.0$ | 6 (7 %) | 9 (11 %) |
| $\lvert z\rvert \ge 3.0$ | 20 (22 %) | 16 (19 %) |
| Total: | 89 (100%) | 85 (100%) |

6. Performance of participants, in terms of zeta-scores, is summarized as follows:

| $\zeta$-score | Number of Participants (Percentage) | |
|---|---|---|
| | Iron | Zinc |
| $\lvert \zeta \rvert \le 2.0$ | 46 (55 %) | 43 (54 %) |
| $2.0 < \lvert \zeta \rvert < 3.0$ | 4 (5 %) | 7 (9 %) |
| $\lvert \zeta \rvert \ge 3.0$ | 33 (40 %) | 29 (37 %) |
| Total: | 83 (100%) | 79 (100%) |

# 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. However, wheat 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. The APMP-APLAC Joint Proficiency Testing (PT) (APLAC PT T100) of toxic elements (lead and cadmium) in wheat flour was conducted in 2015-2016. Wheat also contains a number of elements classified 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. With increasing international trade of food and agricultural products, traceable measurements of elements in agricultural products have become one of the essential requirements for ensuring human health.

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 [4]. The Korea Research Institute of Standards and Science (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 nutritional elements (iron and zinc) in wheat flour. The purpose of this study is to demonstrate the capability of participating laboratories in measuring the contents of iron and zinc at mg/kg levels in a 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 iron and zinc in the test sample were 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

National Metrology Institutes (NMIs) with appropriate CMCs as PT reference values for this APMP-APLAC Joint PTs allows 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. The 113 laboratories were nominated, 100 out of 113 laboratories from 45 economies enrolled in the program and 90 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 (T105-001 to T105-113) and the codes were used throughout the program.

## 2. Measurands

2.1. Mass fractions of iron and zinc in wheat flour on a dry mass.

## 3. Objectives

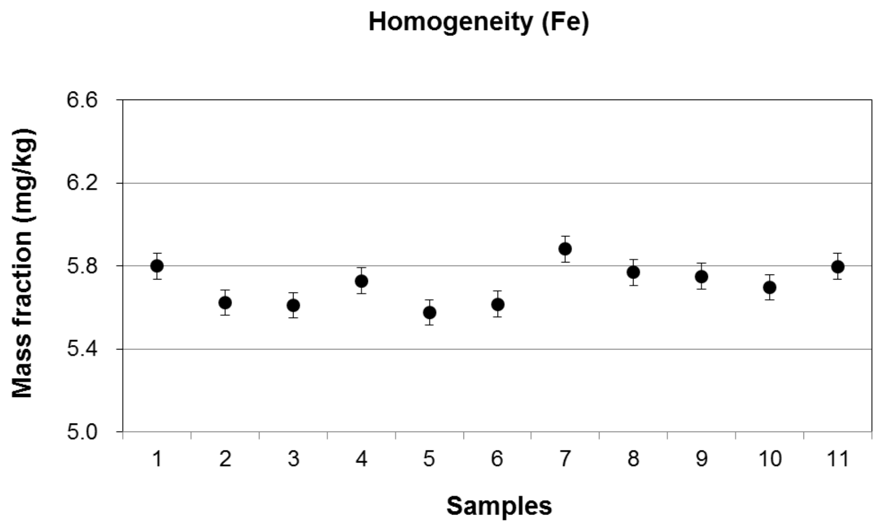
3.1. The aim of this study is to demonstrate the capability of participating laboratories in measuring the amount of iron and zinc in the test sample of wheat flour by various analytical techniques.

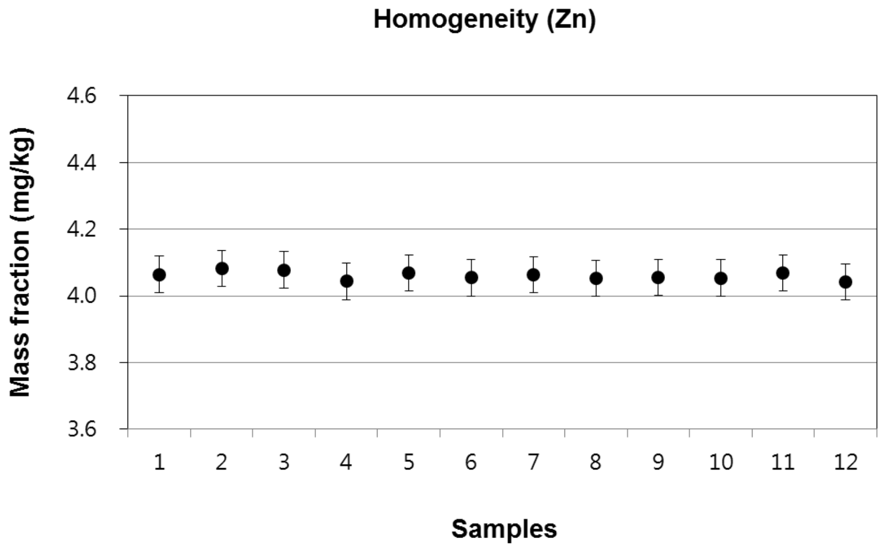
3.2. The design of the PT followed the requirements in ISO/IEC 17043:2010 Conformity Assessment – General Requirements for Proficiency Testing [1] and the test results submitted by participants were processed in accordance with the statistical techniques outlined in ISO13528:2015 Statistical Methods 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 element 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 wheat flour was 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. Freeze-dried wheat flour was ground using a laboratory mill (Pulvurisette 14; Fritsch, Idar-Oberstein, Germany) with 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) 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 Fe and Zn contents due to between-sample inhomogeneity were less than 1.71 % and 0.31 %, respectively. The between-samples standard deviation ($s_s$) was compared with the standard deviation for the proficiency assessment ($\sigma_{pt}$) and found to meet the requirement, $s_s \leq 0.3\sigma_{pt}$, both for Fe and Zn. 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 (Fe and Zn) are shown in the following figures.




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 a rice powder CRM (such as KRISS CRM 108-01-004) 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 PT based on the previous stability studies of KRISS.

4.4. One bottle containing about 20 g of the wheat flour was dispatched to each participant by express mail on 23 August 2016. 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. 82 laboratories returned their sample receipt form.

## 5. Test and Reporting

5.1. Participants were requested to determine the mass fraction (in mg/kg) of iron and zinc 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 (aplacpt.inorg@kriss.re.kr) before the deadline on 21 October 2016.

## 6. Assigned Values for Proficiency Assessment

6.1. The reference value certified by KRISS was used as the assigned value, proficiency test reference value (PTRV), for each element. KRISS used a robust primary method, the double ID-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 PT schemes.

6.2. The assigned values ($x$), standard uncertainties ($u_x$), coverage factors ($k$) and expanded uncertainties ($U_x$) 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) |
|---|---|---|---|---|
| Iron | 5.71 | 0.12 | 2.12 | 0.25 |
| Zinc | 4.06 | 0.06 | 1.96 | 0.11 |

The uncertainties due to the random and systematic effects were combined to get the combined uncertainty [5]. The uncertainty due to the random effects was from the repeatability/reproducibility of the measurements and sample homogeneity. The uncertainty due to the systematic effects was mainly from characterization including uncertainty sources such as calibration of balances, isotopic ratios of isotopes used for double Isotope Dilution Mass Spectrometry (IDMS), amount contents of primary reference solutions, procedure blank corrections, etc. [6]. The uncertainty due to the stability was not considered because it is well known that the elements, Zn and Fe, are very stable in this type of CRM matrix after sterilization by gamma-ray irradiation. The standard uncertainty was estimated by combining the standard deviations due to between-sample variations (relatively 1.71 % and 0.31 % for Fe and Zn, respectively) as random effects and uncertainty contribution from the systematic effects (relatively 1.09 % and 1.34 % for Fe and Zn, respectively).

## 7. Evaluation of Performance of Participants

7.1. Participants' performance was assessed using the *z*-score, ζ-score and $E_n$ score, which are calculated based on Equations (1), (2) and (3), respectively, as follows:

$$z_i = \frac{x_i - x_{pt}}{\sigma_{pt}} \quad (1)$$

where $x_i$ = the reported result of the individual participant
$x_{pt}$ = the assigned value
$\sigma_{pt}$ = 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 = $10^{-6}$)]

$$\zeta_i = \frac{x_i - x_{pt}}{\sqrt{u_{x_i}^2 + u_{x_{pt}}^2}} \quad (2)$$

where $u_{xi}$ = the estimated standard uncertainty of $x_i$ based on the reported results of individual participant
$u_{xpt}$ = the standard uncertainty of $x_{pt}$

$$E_{n_i} = \frac{x_i - x_{pt}}{\sqrt{U_{x_i}^2 + U_{x_{pt}}^2}} \quad (3)$$

where $U_{xi}$ = the estimated expanded uncertainty of $x_i$ based on the reported results of individual participant
$U_{xpt}$ = the expanded uncertainty of $x_{pt}$

7.2. The *z*-score and ζ-score are commonly interpreted as:

(i) |score| ≤ 2.0 — Satisfactory

(ii) 2.0 < |score| < 3.0 — Questionable

(iii) |score| ≥ 3.0 — Unsatisfactory

In case of $E_n$ score, -1.0 < $E_n$ < 1.0 should be taken as an indicator of satisfactory performance.

7.3. Laboratories having |score| ≥ 3.0 should thoroughly investigate their results for the discrepancy and those having *z*-scores in the range 2.0 < |score| < 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 20 January 2017. Sixty 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. Ninety participants submitted their results. Eighty nine iron results and eighty five zinc results were evaluated. Eighty five out of all participants submitted both results.

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

| | Iron | Zinc |
|---|---|---|
| No. of Results | 89 | 85 |
| Assigned value (PTRV) | 5.71 mg/kg | 4.06 mg/kg |
| Average of Participants' Results | 8.22 mg/kg | 4.65 mg/kg |
| Difference of the Average from PTRV | 2.51 mg/kg | 0.59 mg/kg |
| Relative Difference of the Average from PTRV | 43.9 % | 14.4 % |
| SD of Participants' Results | 9.86 mg/kg | 1.74 mg/kg |
| Relative SD of Participants' Results | 120 % | 37.4 % |
| Robust Average of Participants' Results | 6.55 mg/kg | 4.35 mg/kg |
| Difference of the Robust Average from PTRV | 0.84 mg/kg | 0.29 mg/kg |
| Relative Difference of the Robust Average from PTRV | 14.7 % | 7.1 % |
| Robust SD | 1.48 mg/kg | 0.93 mg/kg |
| Relative Robust SD | 22.6 % | 21.4 % |
| Median of Participants' Results | 6.18 mg/kg | 4.20 mg/kg |
| Difference of the median from PTRV | 0.47 mg/kg | 0.14 mg/kg |

| | | |
|---|---|---|
| Relative Difference of the median from PTRV | 8.3 % | 3.4 % |
| Scaled median absolute deviation MAD*e* | 1.01 mg/kg | 0.53 mg/kg |
| Relative MAD*e* | 16.4 % | 12.7 % |
| SD Estimated from the Horwitz Equation | 0.70 mg/kg | 0.53 mg/kg |
| Relative Horwitz SD | 12.3 % | 13.0 % |

8.4. Proficiency test reference values (PTRV), the certified values provided by KRISS, for iron and zinc in the sample were used as the assigned values to evaluate the performance of the participants. We observed significant difference of the average of the participants' results with the assigned values for iron results. The relative difference of the average from the assigned value was 43.9 % and 14.4 % for iron and zinc results, respectively. The robust averages, which were derived using the algorithm A in ISO 13528 [2], are 14.7 % and 7.1 % higher and the medians of participants' results are 8.3 % and 3.4 % higher than the corresponding PTRVs for iron and zinc, respectively. Both robust averages and medians are higher than PTRVs, indicating the significant bias of participants' results on the quantitative analysis of iron and zinc. This fact confirms significant importance of the use of metrologically traceable PTRVs as PT assigned values for rigorous evaluation and reliable assessment. For both iron and zinc, the each standard deviation obtained from the participants' results is much larger than corresponding $\sigma_H$ (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 *z*-scores are calculated by adopting $\sigma_H$ as the expected standard deviation of this study, which was announced in the study protocol. In fact, Horwitz Equation is generally accepted in many PT 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 both *z*-score and zeta score. The patterns of *z*-score distributions in ascending order, for all participants, are shown in FIGUREs I and II for iron and zinc, respectively. The patterns of zeta score distributions are also shown FIGUREs III and IV. The results with *z*-scores and zeta scores marked bold with blue colour or red colour were considered as questionable (i.e. 2.0 < |score| < 3.0) or unsatisfactory (i.e. |score| ≥ 3.0), respectively. The results with $|E_n| \geq 1.0$ are marked bold with red colour. The standard uncertainty and expanded uncertainty reported from each participant were used to derive zeta-scores and $E_n$ scores, respectively. $E_n$ scores are provided just for information. The zeta-scores and $E_n$ scores of the reported results were calculated following ISO 13528. The zeta scores and $E_n$ scores are effective for the assessment of the reported results considering their estimated standard uncertainties and expanded uncertainties.

8.6. The comparisons between the assigned values and the participants' reported results with associated expanded uncertainties, if provided, are also presented in FIGUREs V and VI.

8.7. Performance of participants, in terms of *z*-scores, is summarized as follows:

| *z*-score | Number of Participants (Percentage) | |
|---|---|---|
| | Iron | Zinc |
| $\|z\| \leq 2.0$ | 63 (71 %) | 60 (71 %) |
| $2.0 < \|z\| < 3.0$ | 6 (7 %) | 9 (11 %) |
| $\|z\| \geq 3.0$ | 20 (22 %) | 16 (19 %) |
| Total: | 89 (100%) | 85 (100%) |

In brief, fifty two participants reported satisfactory results with $|z| \leq 2.0$ for both analytes. Ten participants (Lab Code: T105-002, 003, 009, 038, 049, 074, 079, 094, 106 and 111) were identified as having unsatisfactory results with $|z| \geq 3.0$ for both analytes and twenty five participants were found to have unsatisfactory results for one of the analytes.

8.8. Performance of participants, in terms of zeta-scores, is summarized as follows:

| $\zeta$-score | Number of Participants (Percentage) | |
|---|---|---|
| | Iron | Zinc |
| $\|\zeta\| \leq 2.0$ | 46 (55 %) | 43 (54 %) |
| $2.0 < \|\zeta\| < 3.0$ | 4 (5 %) | 7 (9 %) |
| $\|\zeta\| \geq 3.0$ | 33 (40 %) | 29 (37 %) |
| Total: | 83 (100%) | 79 (100%) |

Thirty three participants reported satisfactory results with $|\zeta| \leq 2.0$ for both analytes. Only twenty nine participants got satisfactory results with both $|z| \leq 2.0$ and $|\zeta| \leq 2.0$ for both analytes.

8.9. From the distributions of the participants' scores (Tables in 8.7 and 8.8 above), it can be seen that more participants got "unsatisfactory results" when the results were assessed by zeta scores. Fifteen and sixteen participants obtained $|z| \leq 2.0$ but $|\zeta| > 2.0$ for iron and zinc analysis, respectively. Zeta scores above 2 or below -2 may be caused by systematically biased measurements or by a poor estimation of the measurement uncertainty by the participants. These results show that it is useful for participants to use both *z*-scores and zeta scores for their performance evaluation.

8.10. Youden graphs showing the results distribution among the participants is shown in FIGUREs VII, VIII, IX, XI, XIII, XV, and XVII. X axis and Y axis indicate *z*-scores (also zeta scores in FIGURE VII) for Fe and Zn. Each point represents a pair of results from a single laboratory. The different coloured symbols identify the state of accreditation, the different instrumental methods, the use of internal standard, digestion technique, correction for recovery, or method validation. If results from different laboratories vary entirely because of random variation, one would expect approximately equal number of points in each quadrant. If systematic effects were the main cause of the variation, one would expect that laboratories with a high (or low) value for one analyte would also tend to obtain a high (or low) value for another analyte. This would lead to a predominance of points in the upper right (or lower left) quadrant.

8.11. FIGUREs X, XII, XIV, XVI, and XVIII are the results for Fe and Zn, which are sorted according to instrumental methods used, use of internal standard, digestion technique, recovery correction and method validation.

8.12. Instrumental methods:

As shown in TABLE VIII, TABLE IX, FIGURE IX, and FIGURE X, all participants employed Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) (or expressed as Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)), ICP/MS, Flame Atomic Absorption Spectrometry (FAAS) or Graphite Furnace Atomic Absorption Spectrometry (GFAAS) for measurements. Over one-third of the participants (37 participants) used ICP-OES (ICP-AES) to quantify the levels of analytes. Twenty seven and twenty eight participants used FAAS to quantify the Fe and Zn analytes, respectively. Seventeen participants and two participants employed ICP-MS and GFAAS to quantify both analytes. The Youden plot and results sorted according to the instrumental methods are shown in FIGUREs IX and X, respectively.

8.13. The use of internal standard:

Thirty two (36 %) and thirty one participants (36 %) applied internal standards to quantify the Fe and Zn analytes, respectively. Among those participants who used internal standards twenty nine (91 %) and thirty participants (97 %) were found having satisfactory results with $|z| \leq 2.0$ on Fe and Zn analytes, respectively. The Youden plot and results sorted according to the use of internal standards are shown in FIGUREs XI and XII. As shown in FIGURE XI, it is found that laboratories have $|z| \leq 2.0$ mostly when participants used internal standards to quantify the analytes. Some large relative deviations from PTRV for Fe or Zn were observed when an internal standard was not used.

8.14. Digestion technique:

Fifty one (57 %) and forty eight participants (56 %) employed microwave-assisted digestion to dissolve samples for Fe and Zn analyses, respectively. Seventeen (19 %) and sixteen participants (19 %) employed wet digestion to dissolve samples to analyse Fe and Zn, respectively. Twenty one (24 %) and twenty participants (24 %) dissolved samples by dry ashing to analyse Fe and Zn, respectively. The Youden plot and results sorted according to their digestion technique are shown in FIGUREs XIII and XIV.

8.15. Correction for recovery:

Only eighteen (20 %) and seventeen participants (20 %) corrected their results for recovery to quantify the Fe and Zn analytes, respectively. The Youden plot and results sorted according to their correction for recovery are shown in FIGUREs XV and XVI.

8.16. Method validation:

Sixty four (72 %) and sixty two participants (73 %) claimed their methods were validated for Fe and Zn analytes, respectively. Among those laboratories who employed method validation twenty (31 %) and fifteen laboratories (24 %) were identified as questionable or unsatisfactory in resulted $z$-scores for Fe and Zn. Youden plot and results sorted according to the employment of method validation processes are shown in FIGUREs XVII and XVIII.

## 9. Additional Observations

9.1. Two participants (Lab code: T105-043 and T105-054) had problems in preparing entry documents for sample delivery.

9.2. Three participants (Lab code: T105-012, T105-030 and T105-071) informed that they will not be able to complete the testing.

9.3. One participant (Lab code: T105-023) informed that they don't have the possibility to participate in the PT.

9.4. Three participants (Lab code: T105-005, T105-036, T105-100) submitted sample receipt form, but didn't report their measurement results.

9.5. One participant (Lab code: T105-075) didn't submit both sample receipt form and result report form.

9.6. One participant (Lab code: T105-081) for iron and three laboratories (Lab code: T105-045, T105-065 and T105-081) for zinc reported the results as a range, not values. The results were not included in evaluation of performance of participants.

9.7. Two participants (Lab code: T105-089 and T105-108) submitted only iron results.

9.8. The 82 of 100 participants returned sample receipt form and 90 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 and APMP-APLAC Joint PT Working Group members 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

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

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**Table I: Geographical distribution of participants**

| No. | ECONOMIES | PARTICIPANTS ENROLLED | RETURNED RESULTS |
|---|---|---|---|
| 1 | Australia | 4 | 4 |
| 2 | U.S.A. | 3 | 2 |
| 3 | Hong Kong | 1 | 1 |
| 4 | P.R.China | 4 | 2 |
| 5 | Lithuania | 1 | 1 |
| 6 | India | 4 | 4 |
| 7 | Sweden | 1 | 1 |
| 8 | Israel | 2 | 2 |
| 9 | Netherlands | 2 | 2 |
| 10 | France | 1 | 1 |
| 11 | Estonia | 1 | 1 |
| 12 | Philippines | 3 | 3 |
| 13 | Singapore | 2 | 2 |
| 14 | Sri Lanka | 2 | 2 |
| 15 | Viet Nam | 4 | 4 |
| 16 | Finland | 2 | 2 |
| 17 | Indonesia | 4 | 4 |
| 18 | Switzerland | 2 | 2 |
| 19 | Japan | 4 | 4 |
| 20 | Italy | 2 | 2 |
| 21 | New Zealand | 4 | 4 |
| 22 | Cyprus | 1 | 1 |
| 23 | Bangladesh | 4 | 4 |
| 24 | Spain | 2 | 2 |
| 25 | Malaysia | 2 | 2 |
| 26 | Egypt | 1 | 1 |
| 27 | Sudan | 2 | 1 |
| 28 | Palestine | 2 | 2 |
| 29 | Sultanate of Oman | 2 | 1 |
| 30 | Jordan | 1 | 0 |
| 31 | Iraq | 2 | 1 |
| 32 | Mexico | 2 | 1 |
| 33 | Colombia | 1 | 1 |
| 34 | Taiwan | 4 | 4 |
| 35 | Austria | 2 | 2 |
| 36 | Mongolia | 3 | 2 |
| 37 | Republic of Korea | 5 | 4 |
| 38 | Mauritius | 1 | 1 |
| 39 | Latvia | 1 | 1 |
| 40 | Ethiopia | 2 | 2 |
| 41 | Republic of Serbia | 2 | 2 |
| 42 | Poland | 2 | 2 |
| 43 | Turkey | 1 | 1 |
| 44 | Portugal | 1 | 1 |
| 45 | Zambia | 1 | 1 |
| | Total: | 100 | 90 |

**Table II: Program timeline**

| PHASE | TIME SCHEDULE |
|---|---|
| Call for nomination | June 2016 |
| Deadline for nomination | 22 July 2016 |
| Deadline for registration | 5 August 2016 |
| Dispatch of samples | 23 August 2016 |
| Deadline for submission of results | 21 October 2016 |
| Statistical analysis of results | November 2016 |
| Issue of interim report | 20 December 2016 |
| Issue of final report | November 2017 |

**Table III: Participants' *z*-scores, zeta-scores, and $E_n$ scores for iron and zinc**

| Lab Code | Fe | | | Zn | | |
|---|---|---|---|---|---|---|
| | *z*-score | *ζ*-score | $E_n$ score | *z*-score | *ζ*-score | $E_n$ score |
| T105-001 | 0.09 | 0.15 | 0.08 | -0.06 | -0.06 | -0.03 |
| T105-002 | **3.51** | **13.11** | **6.55** | **11.58** | **38.12** | **19.06** |
| T105-003 | **18.81** | **41.86** | --- | **3.97** | **4.61** | --- |
| T105-004 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-005 | — | — | — | — | — | — |
| T105-006 | 0.70 | 0.27 | 0.14 | 0.55 | 0.63 | 0.32 |
| T105-007 | **9.21** | **5.87** | **2.93** | 0.19 | 0.32 | 0.16 |
| T105-008 | 0.63 | 1.52 | 0.76 | -1.01 | **-4.19** | **-2.09** |
| T105-009 | **8.52** | **13.10** | **6.55** | **3.59** | **11.83** | **5.91** |
| T105-010 | **3.83** | **19.34** | **9.67** | 0.65 | **3.36** | **1.68** |
| T105-011 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-012 | — | — | — | — | — | — |
| T105-013 | 1.29 | **2.17** | **1.11** | **4.26** | **13.24** | **6.81** |
| T105-014 | **2.06** | 1.64 | 0.82 | 0.93 | 1.37 | 0.68 |
| T105-015 | 1.25 | **5.26** | **2.63** | **2.15** | **5.83** | **2.91** |
| T105-016 | -0.87 | -1.18 | -0.59 | -0.21 | -0.43 | -0.21 |
| T105-017 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-018 | -0.68 | -1.47 | -0.74 | -0.07 | -0.18 | -0.09 |
| T105-019 | -1.86 | -0.07 | -0.04 | **-2.40** | -0.17 | -0.09 |
| T105-020 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-021 | 1.48 | **8.23** | **4.11** | -1.67 | **-15.96** | **-7.98** |
| T105-022 | -1.94 | **-9.81** | **-4.71** | **-2.47** | **-20.75** | **-8.74** |
| T105-023 | — | — | — | — | — | — |
| T105-024 | 1.52 | **3.11** | **1.15** | 0.86 | **2.39** | 0.88 |
| T105-025 | **4.41** | **10.11** | **5.05** | -0.36 | -1.54 | -0.77 |
| T105-026 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-027 | -0.16 | -0.26 | -0.13 | -0.30 | -0.90 | -0.45 |
| T105-028 | -1.42 | **-6.91** | **-3.45** | **13.84** | **85.50** | **42.75** |
| T105-029 | 0.26 | 1.21 | 0.61 | 0.00 | 0.00 | 0.00 |
| T105-030 | — | — | — | — | — | — |
| T105-031 | 0.46 | --- | --- | 2.24 | --- | --- |
| T105-032 | **4.40** | **3.48** | **1.70** | 1.22 | 1.30 | 0.64 |
| T105-033 | 0.65 | 0.67 | 0.32 | -0.19 | -0.24 | -0.12 |

| Lab Code | Fe | | | Zn | | |
|---|---|---|---|---|---|---|
| | *z*-score | *ζ*-score | $E_n$ score | *z*-score | *ζ*-score | $E_n$ score |
| T105-034 | 0.77 | 1.76 | 0.88 | -0.84 | **-3.58** | **-1.67** |
| T105-035 | -0.81 | -1.09 | -0.55 | -0.97 | -1.48 | -0.75 |
| T105-036 | — | — | — | — | — | — |
| T105-037 | 0.41 | 0.62 | 0.31 | 1.41 | 1.71 | 0.82 |
| T105-038 | **16.05** | **83.80** | **41.90** | **12.81** | **69.43** | **34.71** |
| T105-039 | 0.70 | **3.30** | **1.62** | 0.95 | **2.30** | **1.15** |
| T105-040 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-041 | 0.69 | **3.00** | **1.50** | 1.23 | **5.56** | **2.77** |
| T105-042 | -0.30 | -0.02 | -0.21 | **-2.15** | -0.10 | **-1.77** |
| T105-043 | — | — | — | — | — | — |
| T105-044 | 1.95 | 1.16 | 0.58 | 0.13 | 0.11 | 0.06 |
| T105-045 | -0.18 | -0.28 | -0.14 | --- | --- | --- |
| T105-046 | 0.18 | 0.35 | 0.17 | 0.02 | 0.04 | 0.02 |
| T105-047 | 1.65 | **8.33** | **4.17** | **-2.01** | **-13.75** | **-6.88** |
| T105-048 | **4.85** | **7.15** | **3.61** | 0.34 | 0.41 | 0.20 |
| T105-049 | **4.51** | **3.08** | **1.54** | **5.63** | **3.56** | **1.77** |
| T105-050 | -0.58 | -1.73 | -0.85 | -1.25 | **-2.84** | **-1.42** |
| T105-051 | 0.92 | --- | --- | 0.80 | --- | --- |
| T105-052 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-053 | -0.16 | -0.25 | -0.13 | -0.27 | -0.43 | -0.22 |
| T105-054 | — | — | — | — | — | — |
| T105-055 | 1.08 | 0.36 | 0.18 | 1.46 | 0.61 | 0.30 |
| T105-056 | 0.39 | 1.98 | 0.99 | -0.83 | **-7.05** | **-3.52** |
| T105-057 | -0.01 | -0.01 | -0.01 | -0.11 | -0.12 | -0.06 |
| T105-058 | -0.44 | -0.18 | -0.09 | -0.11 | -0.23 | -0.12 |
| T105-059 | -0.92 | -1.13 | -0.57 | 0.06 | 0.06 | 0.03 |
| T105-060 | -0.35 | -0.03 | -0.17 | 0.01 | 0.00 | 0.01 |
| T105-061 | -0.78 | **-2.71** | **-1.35** | -0.80 | **-2.23** | **-1.12** |
| T105-062 | -0.30 | -0.23 | -0.12 | -0.49 | -0.43 | -0.24 |
| T105-063 | -0.23 | -0.76 | -0.38 | 1.62 | **3.45** | **1.73** |
| T105-064 | 0.04 | 0.12 | 0.06 | 0.19 | 0.63 | 0.31 |
| T105-065 | 0.15 | 0.34 | 0.17 | --- | --- | --- |
| T105-066 | 0.27 | 0.25 | 0.12 | 0.29 | 0.28 | 0.14 |
| T105-067 | -1.51 | **-6.93** | **-3.44** | **5.78** | **28.59** | **14.41** |

| Lab Code | Fe | | | Zn | | |
|---|---|---|---|---|---|---|
| | *z*-score | *ζ*-score | $E_n$ score | *z*-score | *ζ*-score | $E_n$ score |
| T105-068 | 1.12 | 1.11 | 0.60 | 0.27 | 0.35 | 0.15 |
| T105-069 | 0.27 | 0.81 | 0.23 | 0.08 | 0.19 | 0.08 |
| T105-070 | **2.65** | **4.24** | **2.15** | **3.31** | **4.53** | **2.30** |
| T105-071 | — | — | — | — | — | — |
| T105-072 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-073 | 0.74 | --- | 0.78 | 0.70 | --- | 0.82 |
| T105-074 | **21.82** | **59.06** | **46.78** | **10.45** | **46.72** | **37.50** |
| T105-075 | — | — | — | — | — | — |
| T105-076 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-077 | **-2.08** | **-6.41** | **-3.20** | **-4.63** | **-4.99** | **-2.50** |
| T105-078 | **3.50** | 1.53 | 0.77 | 1.20 | 1.33 | 0.67 |
| T105-079 | **127.40** | --- | --- | **-5.02** | --- | --- |
| T105-080 | 0.53 | 0.27 | 0.14 | -1.84 | -1.31 | -0.65 |
| T105-081 | --- | --- | --- | --- | --- | --- |
| T105-082 | **2.65** | **3.98** | **1.97** | **-2.47** | **-7.28** | **-3.74** |
| T105-083 | -0.44 | -1.59 | -0.79 | -1.10 | **-2.56** | **-1.28** |
| T105-084 | -0.90 | -0.97 | -0.48 | -0.19 | -0.20 | -0.10 |
| T105-085 | **7.06** | **5.29** | **2.64** | 0.93 | 1.11 | 0.55 |
| T105-086 | **10.58** | **56.64** | **28.32** | **2.96** | **25.23** | **12.62** |
| T105-087 | 1.78 | **7.74** | **3.87** | 0.88 | **4.12** | **2.06** |
| T105-088 | 0.70 | 1.47 | 0.73 | 0.29 | 0.69 | 0.35 |
| T105-089 | -0.09 | -0.07 | -0.04 | --- | --- | --- |
| T105-090 | **2.40** | **2.38** | **1.19** | 0.84 | 0.97 | 0.49 |
| T105-091 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-092 | 0.27 | --- | 0.20 | -0.21 | --- | -0.18 |
| T105-093 | 0.14 | 0.79 | 0.38 | -0.61 | **-4.54** | **-1.46** |
| T105-094 | **8.08** | **4.13** | **2.06** | **3.00** | **2.71** | **1.36** |
| T105-095 | 0.94 | **3.95** | **1.97** | 0.44 | 1.42 | 0.71 |
| T105-096 | 0.33 | 0.44 | 0.22 | 0.17 | 0.19 | 0.09 |
| T105-097 | 0.67 | 1.18 | 0.58 | -0.02 | -0.03 | -0.02 |
| T105-098 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-099 | 0.13 | 0.33 | 0.16 | 0.84 | **2.75** | **1.34** |
| T105-100 | — | — | — | — | — | — |

| Lab Code | Fe | | | Zn | | |
|---|---|---|---|---|---|---|
| | *z*-score | *ζ*-score | $E_n$ score | *z*-score | *ζ*-score | $E_n$ score |
| T105-101 | 1.21 | 1.27 | 0.64 | 6.03 | **5.44** | **2.63** |
| T105-102 | **4.25** | --- | --- | 0.46 | --- | --- |
| T105-103 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-104 | 0.38 | **2.06** | **1.03** | 0.15 | 1.28 | 0.64 |
| T105-105 | N/A | N/A | N/A | N/A | N/A | N/A |
| T105-106 | **3.46** | **7.69** | **3.79** | **10.63** | **15.78** | **8.00** |
| T105-107 | -0.27 | -0.42 | -0.22 | 0.17 | 0.27 | 0.14 |
| T105-108 | **2.09** | **9.18** | **4.59** | --- | --- | --- |
| T105-109 | -0.01 | -0.03 | -0.02 | 0.27 | 0.64 | 0.32 |
| T105-110 | **10.13** | **4.30** | **2.15** | **2.26** | 1.67 | 0.84 |
| T105-111 | **20.12** | **113.10** | **56.55** | **3.08** | **29.37** | **14.69** |
| T105-112 | 1.28 | **3.90** | **1.95** | 0.36 | 1.08 | 0.54 |
| T105-113 | 0.31 | 1.63 | 0.82 | 0.63 | **5.27** | **2.63** |

"—" The registered participant did not submit the results.
"---" Data or information was not provided.
N/A: nominated but not registered laboratory
* Lab T105-045, T105-065 and T105-081 reported the range of value for either one of analyte or both.

**Table IV: Participants' results and reported uncertainties for iron**

| Lab Code | Moisture content (%) of the sample | Mass fraction of iron (mg/kg) | Combined standard uncertainty (mg/kg) | Coverage factor, *k* (95% level of confidence) | Expanded uncertainty (mg/kg) |
|---|---|---|---|---|---|
| T105-001 | 0.37 | 5.77 | 0.37 | 2 | 0.74 |
| T105-002 | 2.52 | 8.18 | 0.141 | 2 | 0.282 |
| T105-003 | 0.38 | 18.93 | 0.29 | --- | --- |
| T105-004 | N/A | N/A | N/A | N/A | N/A |
| T105-005 | — | — | — | — | — |
| T105-006 | 98.5 | 6.2 | 1.8 | 2 | 3.6 |
| T105-007 | 2 | 12.1803 | 1.096 | 2 | 2.192 |
| T105-008 | --- | 6.15 | 0.261 | 2 | 0.523 |
| T105-009 | 1.07 | 11.7 | 0.44 | 2 | 0.88 |
| T105-010 | | 8.4 | 0.061 | 2 | 0.122 |
| T105-011 | N/A | N/A | N/A | N/A | N/A |
| T105-012 | — | — | — | — | — |
| T105-013 | 2.4 | 6.62 | 0.4 | 1.96 | 0.78 |
| T105-014 | 1.22 | 7.16 | 0.875 | 2 | 1.75 |
| T105-015 | 1.718 | 6.586 | 0.11 | 2 | 0.22 |
| T105-016 | 13 | 5.1 | 0.5 | 2 | 1 |
| T105-017 | N/A | N/A | N/A | N/A | N/A |
| T105-018 | 1.21 | 5.231 | 0.3 | 2 | 0.6 |
| T105-019 | 5.5 | 4.4 | 19 | 1.96 | 37.2 |
| T105-020 | N/A | N/A | N/A | N/A | N/A |
| T105-021 | 1.96 | 6.75 | 0.019 | 2 | 0.038 |
| T105-022 | 2.22 | 4.35 | 0.06 | 2.306 | 0.145 |
| T105-023 | — | — | — | — | — |
| T105-024 | 1.5 | 6.78 | 0.32 | 2 | 0.9 |
| T105-025 | 9.0 | 8.81 | 0.28 | 2 | 0.56 |
| T105-026 | N/A | N/A | N/A | N/A | N/A |
| T105-027 | --- | 5.6 | 0.4 | 2 | 0.8 |
| T105-028 | 1 | 4.71 | 0.073 | 2 | 0.146 |
| T105-029 | 0.58 | 5.89 | 0.08 | 2 | 0.16 |
| T105-030 | — | — | — | — | — |
| T105-031 | 6.8 | 6.03 | --- | --- | --- |
| T105-032 | 1.44 | 8.8 | 0.88 | 2 | 1.8 |
| T105-033 | 1.1 | 6.17 | 0.68 | 2 | 1.4 |

| Lab Code | Moisture content (%) of the sample | Mass fraction of iron (mg/kg) | Combined standard uncertainty (mg/kg) | Coverage factor, *k* (95% level of confidence) | Expanded uncertainty (mg/kg) |
|---|---|---|---|---|---|
| T105-034 | 2.3 | 6.25 | 0.28 | 2 | 0.56 |
| T105-035 | 0.3 | 5.14 | 0.51 | 2 | 1 |
| T105-036 | — | — | — | — | — |
| T105-037 | 1.24 | 6 | 0.45 | 2 | 0.9 |
| T105-038 | --- | 16.992 | 0.05 | 2 | 0.1 |
| T105-039 | 3.2 | 6.2 | 0.08 | 2 | 0.17 |
| T105-040 | N/A | N/A | N/A | N/A | N/A |
| T105-041 | 0.0047 | 6.192 | 0.101 | 2 | 0.201 |
| T105-042 | 2.68 | 5.5 | 8.834 | 2 | 0.95 |
| T105-043 | — | — | — | — | — |
| T105-044 | 3.22 | 7.08 | 1.17 | 2 | 2.34 |
| T105-045 | 1.69(1); 2.21(2); 2.30(3) | 5.58 | 0.447 | 2 | 0.894 |
| T105-046 | 1.42 | 5.84 | 0.35 | 2 | 0.7 |
| T105-047 | 2.61 | 6.867 | 0.0607 | 2 | 0.121 |
| T105-048 | 1.21 | 9.12 | 0.46 | 2 | 0.91 |
| T105-049 | 2.4 | 8.88 | 1.02 | 2 | 2.04 |
| T105-050 | --- | 5.3 | 0.201 | 0.201 | 0.412 |
| T105-051 | --- | 6.36 | --- | --- | --- |
| T105-052 | N/A | N/A | N/A | N/A | N/A |
| T105-053 | 0.5 | 5.6 | 0.42 | 2 | 0.84 |
| T105-054 | — | — | — | — | — |
| T105-055 | 3.40 | 6.47 | 2.12 | 2 | 4.25 |
| T105-056 | --- | 5.987 | 0.063 | 2 | 0.125 |
| T105-057 | 1.4 | 5.7 | 0.7 | 2 | 1.4 |
| T105-058 | 0.5492 | 5.4 | 1.75 | 2 | 3.5 |
| T105-059 | 1.12 | 5.06 | 0.56 | 2 | 1.11 |
| T105-060 | 2.6 | 5.464 | 7.557 | 0.187 | 1.416 |
| T105-061 | 1.8 | 5.16 | 0.16 | 2 | 0.32 |
| T105-062 | 0.31 | 5.5 | 0.9 | 2 | 1.7 |
| T105-063 | 0.355 | 5.55 | 0.17 | 2 | 0.34 |
| T105-064 | 0.134 | 5.74 | 0.22 | 2 | 0.44 |
| T105-065 | 2.00 | 5.8163 | 0.2908 | 2 | 0.5816 |

| Lab Code | Moisture content (%) of the sample | Mass fraction of iron (mg/kg) | Combined standard uncertainty (mg/kg) | Coverage factor, *k* (95% level of confidence) | Expanded uncertainty (mg/kg) |
|---|---|---|---|---|---|
| T105-066 | 0.87 | 5.9 | 0.76 | 2 | 1.52 |
| T105-067 | | 4.65 | 0.088 | 2 | 0.18 |
| T105-068 | 1.81 | 6.5 | 0.7 | 2 | 1.3 |
| T105-069 | < 1.0 | 5.9 | 0.2 | 2 | 0.8 |
| T105-070 | 99.4 | 7.57 | 0.42 | 2 | 0.83 |
| T105-071 | — | — | — | — | — |
| T105-072 | N/A | N/A | N/A | N/A | N/A |
| T105-073 | 1.33 | 6.23 | --- | --- | 0.62 |
| T105-074 | --- | 21.0445 | 0.22756517 | 18.8789-23.9211 | 0.21197717 |
| T105-075 | — | — | — | — | — |
| T105-076 | N/A | N/A | N/A | N/A | N/A |
| T105-077 | 1.74685 | 4.2472 | 0.191 | 2 | 0.382 |
| T105-078 | 99.31 | 8.17 | 1.6 | 2 | 3.2 |
| T105-079 | 1.3 | 95.24 | --- | 0.76 | --- |
| T105-080 | 4.85 | 6.08 | 1.34 | 2 | 2.68 |
| T105-081 | --- | <10.00 | --- | --- | --- |
| T105-082 | 0.96 | 7.57 | 0.45 | 2 | 0.91 |
| T105-083 | No | 5.4 | 0.15 | 2 | 0.3 |
| T105-084 | NO | 5.08 | 0.64 | 2 | 1.28 |
| T105-085 | 0.25 | 10.67 | 0.93 | 2 | 1.86 |
| T105-086 | 0.03 | 13.1441 | 0.04 | 0.0089/2 | 0.08 |
| T105-087 | 3.0159 | 6.9624 | 0.1028 | 2 | 0.2056 |
| T105-088 | --- | 6.2 | 0.31 | 2 | 0.62 |
| T105-089 | 0.9 | 5.65 | 0.792 | 2 | 1.58 |
| T105-090 | 1.64 | 7.4 | 0.7 | 2 | 1.4 |
| T105-091 | N/A | N/A | N/A | N/A | N/A |
| T105-092 | 0.5 | 5.9 | --- | --- | 0.9 |
| T105-093 | 1.33 | 5.81 | 0.02 | 4.302 | 0.09 |
| T105-094 | 0.84 | 11.39 | 1.37 | 2 | 2.74 |
| T105-095 | 7.4 | 6.37 | 0.111 | 2 | 0.222 |
| T105-096 | 2.208 | 5.94 | 0.51 | 2 | 1.02 |
| T105-097 | 7.63 | 6.182 | 0.38 | 2 | 0.77 |
| T105-098 | N/A | N/A | N/A | N/A | N/A |

| Lab Code | Moisture content (%) of the sample | Mass fraction of iron (mg/kg) | Combined standard uncertainty (mg/kg) | Coverage factor, *k* (95% level of confidence) | Expanded uncertainty (mg/kg) |
|---|---|---|---|---|---|
| T105-099 | 1.7 | 5.8 | 0.24 | 0.24 | 0.49 |
| T105-100 | — | — | — | — | — |
| T105-101 | 0,6 | 6.56 | 0.66 | 2 | 1.3 |
| T105-102 | 2.41 | 8.7 | --- | --- | --- |
| T105-103 | N/A | N/A | N/A | N/A | N/A |
| T105-104 | 1.40 | 5.98 | 0.04 | 2 | 0.08 |
| T105-105 | N/A | N/A | N/A | N/A | N/A |
| T105-106 | 0.185 | 8.14 | 0.29 | 2 | 0.59 |
| T105-107 | 2.85 | 5.52 | 0.43 | 1.96 | 0.84 |
| T105-108 | 3.55 | 7.18 | 0.1 | 2 | 0.2 |
| T105-109 | 1.4 | 5.7 | 0.29 | 2 | 0.57 |
| T105-110 | 2.8 | 12.83 | 1.65 | 2 | 3.3 |
| T105-111 | 0.37 | 19.85 | 0.00254846 | 1.96 | 0.00499498 |
| T105-112 | 0.995 | 6.6129 | 0.195 | 2 | 0.389 |
| T105-113 | 1.41 | 5.93 | 0.05 | 2 | 0.1 |

"—" The registered participant did not submit the results.
"---" Data or information was not provided.
N/A: nominated but not registered laboratory
* Lab T105-081 reported the range of value.

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

| Lab Code | Mass fraction of zinc (mg/kg) | Combined standard uncertainty (mg/kg) | Coverage factor, *k* (95% level of confidence) | Expanded uncertainty (mg/kg) | Laboratory accreditation (Yes/No) |
|---|---|---|---|---|---|
| T105-001 | 4.03 | 0.46 | 2 | 0.91 | YES |
| T105-002 | 10.15 | 0.15 | 2 | 0.3 | YES |
| T105-003 | 6.15 | 0.45 | --- | --- | YES |
| T105-004 | N/A | N/A | N/A | N/A | N/A |
| T105-005 | — | — | — | — | — |
| T105-006 | 4.35 | 0.46 | 2 | 0.91 | YES |
| T105-007 | 4.1601 | 0.312 | 2 | 0.624 | YES |
| T105-008 | 3.53 | 0.114 | 2 | 0.229 | YES |
| T105-009 | 5.95 | 0.15 | 2 | 0.3 | YES |
| T105-010 | 4.4 | 0.085 | 2 | 0.17 | YES |
| T105-011 | N/A | N/A | N/A | N/A | N/A |
| T105-012 | — | — | — | — | — |
| T105-013 | 6.3 | 0.16 | 1.96 | 0.3100 | YES |
| T105-014 | 4.55 | 0.353 | 2 | 0.707 | YES |
| T105-015 | 5.19 | 0.186 | 2 | 0.372 | YES |
| T105-016 | 3.95 | 0.25 | 2 | 0.5 | YES |
| T105-017 | N/A | N/A | N/A | N/A | N/A |
| T105-018 | 4.023 | 0.2 | 2 | 0.4 | --- |
| T105-019 | 2.8 | 7.5 | 1.96 | 14.7 | NO |
| T105-020 | N/A | N/A | N/A | N/A | N/A |
| T105-021 | 3.18 | 0.0039 | 2 | 0.0078 | YES |
| T105-022 | 2.76 | 0.03 | 3.182 | 0.1 | YES |
| T105-023 | — | — | — | — | — |
| T105-024 | 4.51 | 0.18 | 2 | 0.5 | YES |
| T105-025 | 3.87 | 0.11 | 2 | 0.22 | YES |
| T105-026 | N/A | N/A | N/A | N/A | N/A |
| T105-027 | 3.9 | 0.17 | 2 | 0.34 | YES |
| T105-028 | 11.34 | 0.065 | 2 | 0.13 | YES |
| T105-029 | 4.06 | 0.09 | 2 | 0.18 | YES |
| T105-030 | — | — | — | — | — |
| T105-031 | 5.24 | --- | --- | --- | YES |

| Lab Code | Mass fraction of zinc (mg/kg) | Combined standard uncertainty (mg/kg) | Coverage factor, *k* (95% level of confidence) | Expanded uncertainty (mg/kg) | Laboratory accreditation (Yes/No) |
|---|---|---|---|---|---|
| T105-032 | 4.7 | 0.49 | 2 | 0.99 | YES |
| T105-033 | 3.96 | 0.42 | 2 | 0.83 | YES |
| T105-034 | 3.62 | 0.11 | 2 | 0.24 | YES |
| T105-035 | 3.55 | 0.34 | 2 | 0.67 | YES |
| T105-036 | — | — | — | — | — |
| T105-037 | 4.8 | 0.43 | 2 | 0.9 | YES |
| T105-038 | 10.8 | 0.08 | 2 | 0.16 | YES |
| T105-039 | 4.56 | 0.21 | 2 | 0.42 | YES |
| T105-040 | N/A | N/A | N/A | N/A | N/A |
| T105-041 | 4.709 | 0.103 | 2 | 0.207 | NO |
| T105-042 | 2.93 | 11.055 | 2 | 0.63 | YES |
| T105-043 | — | — | — | — | — |
| T105-044 | 4.13 | 0.62 | 2 | 1.12 | YES |
| T105-045 | <5 | --- | --- | --- | YES |
| T105-046 | 4.07 | 0.24 | 2 | 0.48 | YES |
| T105-047 | 3.004 | 0.0536 | 2 | 0.107 | YES |
| T105-048 | 4.24 | 0.44 | 2 | 0.88 | YES |
| T105-049 | 7.02 | 0.83 | 2 | 1.67 | YES |
| T105-050 | 3.4 | 0.226 | 0.226 | 0.452 | YES |
| T105-051 | 4.48 | --- | --- | --- | YES |
| T105-052 | N/A | N/A | N/A | N/A | N/A |
| T105-053 | 3.92 | 0.32 | 2 | 0.64 | YES |
| T105-054 | — | — | — | — | — |
| T105-055 | 4.83 | 1.27 | 2 | 2.54 | NO |
| T105-056 | 3.625 | 0.028 | 2 | 0.056 | YES |
| T105-057 | 4 | 0.5 | 2 | 1 | YES |
| T105-058 | 4 | 0.26 | 2 | 0.5 | YES |
| T105-059 | 4.09 | 0.49 | 2 | 0.98 | YES |
| T105-060 | 4.066 | 8.949 | 0.085 | 0.765 | YES |
| T105-061 | 3.64 | 0.18 | 2 | 0.36 | YES |
| T105-062 | 3.8 | 0.6 | 2 | 1.1 | YES |
| T105-063 | 4.91 | 0.24 | 2 | 0.48 | NO |
| T105-064 | 4.16 | 0.15 | 2 | 0.3 | YES |

| Lab Code | Mass fraction of zinc (mg/kg) | Combined standard uncertainty (mg/kg) | Coverage factor, *k* (95% level of confidence) | Expanded uncertainty (mg/kg) | Laboratory accreditation (Yes/No) |
|---|---|---|---|---|---|
| T105-065 | Less than 5 | 0.25 | 2 | 0.5 | YES |
| T105-066 | 4.21 | 0.54 | 2 | 1.08 | YES |
| T105-067 | 7.1 | 0.091 | 2 | 0.18 | YES |
| T105-068 | 4.2 | 0.4 | 2 | 0.9 | YES |
| T105-069 | 4.1 | 0.2 | 2 | 0.5 | YES |
| T105-070 | 5.8 | 0.38 | 2 | 0.750 | YES |
| T105-071 | — | — | — | — | — |
| T105-072 | N/A | N/A | N/A | N/A | N/A |
| T105-073 | 4.43 | --- | --- | 0.44 | YES |
| T105-074 | 9.55553 | 0.10397789 | 6.1515-12.9152 | 0.0968555 | NO |
| T105-075 | — | — | — | — | — |
| T105-076 | N/A | N/A | N/A | N/A | N/A |
| T105-077 | 1.6225 | 0.485 | 2 | 0.97 | NO |
| T105-078 | 4.69 | 0.47 | 2 | 0.94 | YES |
| T105-079 | 1.42 | --- | 0.2 | --- | NO |
| T105-080 | 3.09 | 0.74 | 2 | 1.48 | YES |
| T105-081 | <10.00 | --- | --- | --- | YES |
| T105-082 | 2.76 | 0.17 | 2 | 0.33 | YES |
| T105-083 | 3.48 | 0.22 | 2 | 0.44 | YES |
| T105-084 | 3.96 | 0.5 | 2 | 1 | YES |
| T105-085 | 4.55 | 0.44 | 2 | 0.88 | YES |
| T105-086 | 5.6172 | 0.028 | 0.00136/2 | 0.056 | --- |
| T105-087 | 4.5249 | 0.0986 | 2 | 0.1972 | YES |
| T105-088 | 4.21 | 0.21 | 2 | 0.42 | YES |
| T105-089 | --- | --- | --- | --- | YES |
| T105-090 | 4.5 | 0.45 | 2 | 0.9 | YES |
| T105-091 | N/A | N/A | N/A | N/A | N/A |
| T105-092 | 3.95 | --- | --- | 0.6 | --- |
| T105-093 | 3.74 | 0.044 | 4.302 | 0.19 | YES |
| T105-094 | 5.64 | 0.58 | 2 | 1.16 | YES |
| T105-095 | 4.29 | 0.152 | 2 | 0.303 | YES |

| Lab Code | Mass fraction of zinc (mg/kg) | Combined standard uncertainty (mg/kg) | Coverage factor, *k* (95% level of confidence) | Expanded uncertainty (mg/kg) | Laboratory accreditation (Yes/No) |
|---|---|---|---|---|---|
| T105-096 | 4.15 | 0.48 | 2 | 0.96 | YES |
| T105-097 | 4.05 | 0.29 | 2 | 0.58 | YES |
| T105-098 | N/A | N/A | N/A | N/A | N/A |
| T105-099 | 4.5 | 0.15 | 0.15 | 0.31 | YES |
| T105-100 | — | — | — | — | — |
| T105-101 | 7.23 | 0.58 | 2 | 1.2 | YES |
| T105-102 | 4.3 | --- | --- | --- | YES |
| T105-103 | N/A | N/A | N/A | N/A | N/A |
| T105-104 | 4.14 | 0.03 | 2 | 0.06 | YES |
| T105-105 | N/A | N/A | N/A | N/A | N/A |
| T105-106 | 9.65 | 0.35 | 2 | 0.69 | YES |
| T105-107 | 4.15 | 0.33 | 1.96 | 0.64 | YES |
| T105-108 | --- | --- | --- | --- | YES |
| T105-109 | 4.2 | 0.21 | 2 | 0.42 | YES |
| T105-110 | 5.25 | 0.71 | 2 | 1.42 | NO |
| T105-111 | 5.679 | 0.003634515 | 1.96 | 0.007123649 | NO |
| T105-112 | 4.2491 | 0.167 | 2 | 0.334 | YES |
| T105-113 | 4.39 | 0.03 | 2 | 0.06 | YES |

"—" The registered participant did not submit the results.
"---" Data or information was not provided.
N/A: nominated but not registered laboratory
* Lab T105-045, T105-065 and T105-081 reported the range of value.

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

| Lab Code | Digestion technique | Digestion medium | Matrix separation? |
|---|---|---|---|
| T105-001 | Closed Vessel/ Hot Block | $HNO_3$/HCl | NO |
| T105-002 | Dry Ashing | $HNO_3$/HCl | --- |
| T105-003 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | YES |
| T105-004 | N/A | N/A | N/A |
| T105-005 | — | — | — |
| T105-006 | Wet digestion | $HNO_3$/HCl | YES |
| T105-007 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-008 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-009 | Dry Ashing | $HNO_3$ | NO |
| T105-010 | Wet digestion | 50% $HNO_3$ and 50% HCl | NO |
| T105-011 | N/A | N/A | N/A |
| T105-012 | — | — | — |
| T105-013 | Microwave-assisted digestion | $HNO_3$/HCl (9.0 ml+1.0 ml) | NO |
| T105-014 | Dry ashing | $HNO_3$/HCl | NO |
| T105-015 | Dry ashing | $HNO_3$ | NO |
| T105-016 | Wet digestion | $HNO_3/HCl/H_2O_2$ | NO |
| T105-017 | N/A | N/A | N/A |
| T105-018 | Microwave-assisted digestion | $HNO_3$ | YES |
| T105-019 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-020 | N/A | N/A | N/A |
| T105-021 | Wet digestion | $HNO_3$ | NO |
| T105-022 | Dry ashing | $HNO_3$ | NO |
| T105-023 | — | — | — |
| T105-024 | Microwave-assisted digestion | $HNO_3$/HCl | NO |
| T105-025 | Dry ashing | HCl | NO |
| T105-026 | N/A | N/A | N/A |
| T105-027 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-028 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-029 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-030 | — | — | — |
| T105-031 | Dry Ashing | $HNO_3$ | Yes |
| T105-032 | Dry Ashing | HCl | --- |
| T105-033 | Microwave-assisted digestion | $HNO_3/HF/H_2O_2$ | NO |

| Lab Code | Digestion technique | Digestion medium | Matrix separation? |
|---|---|---|---|
| T105-034 | Microwave-assisted digestion | $HNO_3$ | YES |
| T105-035 | Wet digestion | $HNO_3$/HCl | NO |
| T105-036 | — | — | — |
| T105-037 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-038 | Wet digestion | Aqua regia | Yes |
| T105-039 | Microwave-assisted digestion | $HNO_3/HClO_4$ | NO |
| T105-040 | N/A | N/A | N/A |
| T105-041 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-042 | Wet digestion | $HNO_3$ | YES |
| T105-043 | — | — | — |
| T105-044 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-045 | Microwave-assisted digestion | $HNO_3$/water | NO |
| T105-046 | Wet digestion | $HNO_3/HClO_4$ | YES |
| T105-047 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-048 | Dry Ashing | HCl | NO |
| T105-049 | Dry Ashing | none | NO |
| T105-050 | Microwave-assisted digestion | $HNO_3/HCl/H_2O_2$ | NO |
| T105-051 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-052 | N/A | N/A | N/A |
| T105-053 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-054 | — | — | — |
| T105-055 | Dry Ashing | $HNO_3$ | YES |
| T105-056 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-057 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-058 | Dry ashing | $HNO_3$ | NO |
| T105-059 | Microwave-assisted digestion | $HNO_3$ / $H_2O_2$ | NO |
| T105-060 | Microwave-assisted digestion | $HNO_3$ | YES |
| T105-061 | Dry ashing | HCl | NO |
| T105-062 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-063 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-064 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | --- |
| T105-065 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-066 | Wet digestion | $HNO_3$/ HF | NO |
| T105-067 | Wet digestion | $HNO_3$ | NO |
| T105-068 | Microwave-assisted digestion | $HNO_3$ | --- |

| Lab Code | Digestion technique | Digestion medium | Matrix separation? |
|---|---|---|---|
| T105-069 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | --- |
| T105-070 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-071 | — | — | — |
| T105-072 | N/A | N/A | N/A |
| T105-073 | Wet digestion | $HNO_3$ | NO |
| T105-074 | Microwave-assisted digestion | $HNO_3/HCl/H_2O_2$/Aqua regia | Yes |
| T105-075 | — | — | — |
| T105-076 | N/A | N/A | N/A |
| T105-077 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-078 | Wet digestion | $HNO_3$ | Yes |
| T105-079 | Wet digestion | $HNO_3/HCl$ | YES |
| T105-080 | Microwave-assisted digestion | $HNO_3$ | |
| T105-081 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-082 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-083 | Dry- Ash Digestion- AOAC 999.11 | --- | --- |
| T105-084 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-085 | Microwave-assisted digestion | $HNO_3/HCl/H_2O_2$ | NO |
| T105-086 | Dry ashing | $HNO_3/HCl$ | --- |
| T105-087 | Dry ashing | $HNO_3$/ HCl | NO |
| T105-088 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-089 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-090 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-091 | N/A | N/A | N/A |
| T105-092 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-093 | Wet digestion | $HNO_3/HCl$ | NO |
| T105-094 | Dry ashing | $HNO_3$ | Yes |
| T105-095 | Wet digestion | $HNO_3/HF$ | NO |
| T105-096 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-097 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-098 | N/A | N/A | N/A |
| T105-099 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-100 | — | — | — |
| T105-101 | Microwave-assisted digestion | $HNO_3/H_2O_2$/Aqua regia | NO |
| T105-102 | Dry ashing | $HNO_3$ | NO |

| Lab Code | Digestion technique | Digestion medium | Matrix separation? |
|---|---|---|---|
| T105-103 | N/A | N/A | N/A |
| T105-104 | Dry ashing | HCl | NO |
| T105-105 | N/A | N/A | N/A |
| T105-106 | Microwave-assisted digestion | $HNO_3$/HCl/$H_2O_2$ | --- |
| T105-107 | Wet digestion | $HNO_3$/ HCl | NO |
| T105-108 | Dry ashing | HCl | NO |
| T105-109 | Microwave digestion | 5% $HNO_3$ | --- |
| T105-110 | Microwave-assisted digestion | $HNO_3$/$H_2O_2$ | --- |
| T105-111 | Microwave-assisted digestion | $HNO_3$ + $H_2O_2$ (7:1) | Yes |
| T105-112 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-113 | Dry ashing | HCl | NO |

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

| Lab Code | Digestion technique | Digestion medium | Matrix separation |
|---|---|---|---|
| T105-001 | Closed Vessel/ Hot Block | $HNO_3/HCl$ | NO |
| T105-002 | Dry Ashing | $HNO_3/HCl$ | --- |
| T105-003 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | YES |
| T105-004 | N/A | N/A | N/A |
| T105-005 | — | — | — |
| T105-006 | Wet digestion | $HNO_3/HCl$ | YES |
| T105-007 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-008 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-009 | Dry Ashing | $HNO_3$ | NO |
| T105-010 | Wet digestion | 50 % $HNO_3$ and 50 % HCl | NO |
| T105-011 | N/A | N/A | N/A |
| T105-012 | — | — | — |
| T105-013 | Microwave-assisted digestion | $HNO_3/HCl$ (9.0 ml+1.0 ml) | NO |
| T105-014 | Dry Ashing | $HNO_3/HCl$ | NO |
| T105-015 | Dry ashing | $HNO_3$ | NO |
| T105-016 | Wet digestion | $HNO_3/HCl/H_2O_2$ | NO |
| T105-017 | N/A | N/A | N/A |
| T105-018 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-019 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-020 | N/A | N/A | N/A |
| T105-021 | Wet digestion | $HNO_3$ | NO |
| T105-022 | Dry ashing | $HNO_3$ | NO |
| T105-023 | — | — | — |
| T105-024 | Microwave-assisted digestion | $HNO_3/HCl$ | NO |
| T105-025 | Dry ashing | HCl | NO |
| T105-026 | N/A | N/A | N/A |
| T105-027 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-028 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-029 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-030 | — | — | — |
| T105-031 | Dry Ashing | $HNO_3$ | YES |
| T105-032 | Dry Ashing | HCl | --- |
| T105-033 | Microwave-assisted digestion | $HNO_3/HF/H_2O_2$ | NO |

| Lab Code | Digestion technique | Digestion medium | Matrix separation |
|---|---|---|---|
| T105-034 | Microwave-assisted digestion | $HNO_3$ | YES |
| T105-035 | Wet digestion | $HNO_3$/HCl | NO |
| T105-036 | — | — | — |
| T105-037 | Microwave-assisted digestion | $HNO_3$/$H_2O_2$ | NO |
| T105-038 | --- | Aqua regia | YES |
| T105-039 | Microwave-assisted digestion | $HNO_3$/$HClO_4$ | NO |
| T105-040 | N/A | N/A | N/A |
| T105-041 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-042 | Wet digestion | $HNO_3$ | YES |
| T105-043 | — | — | — |
| T105-044 | Microwave-assisted digestion | $HNO_3$/$H_2O_2$ | NO |
| T105-045 | Microwave-assisted digestion | $HNO_3$/water | NO |
| T105-046 | Wet digestion | $HNO_3$/$HClO_4$ | YES |
| T105-047 | Microwave-assisted digestion | $HNO_3$/$H_2O_2$ | --- |
| T105-048 | Dry Ashing | HCl | NO |
| T105-049 | Dry Ashing | No | NO |
| T105-050 | Microwave-assisted digestion | $HNO_3$/HCl/$H_2O_2$ | NO |
| T105-051 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-052 | N/A | N/A | N/A |
| T105-053 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-054 | — | — | — |
| T105-055 | Dry Ashing | $HNO_3$ | YES |
| T105-056 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-057 | Microwave-assisted digestion | $HNO_3$/$H_2O_2$ | NO |
| T105-058 | Dry ashing | $HNO_3$ | NO |
| T105-059 | Microwave-assisted digestion | $HNO_3$/$H_2O_2$ | NO |
| T105-060 | Microwave-assisted digestion | $HNO_3$ | YES |
| T105-061 | Dry ashing | HCl | NO |
| T105-062 | Microwave-assisted digestion | $HNO_3$/$H_2O_2$ | NO |
| T105-063 | Microwave-assisted digestion | $HNO_3$/$H_2O_2$ | NO |
| T105-064 | Microwave-assisted digestion | $HNO_3$/$H_2O_2$ | YES |
| T105-065 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-066 | Wet digestion | $HNO_3$/HF | NO |
| T105-067 | Wet digestion | $HNO_3$/$H_2SO_4$ | NO |
| T105-068 | Microwave-assisted digestion | $HNO_3$ | --- |

| Lab Code | Digestion technique | Digestion medium | Matrix separation |
|---|---|---|---|
| T105-069 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | --- |
| T105-070 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-071 | — | — | — |
| T105-072 | N/A | N/A | N/A |
| T105-073 | Wet digestion | $HNO_3$ | NO |
| T105-074 | Microwave-assisted digestion | $HNO_3/HCl/H_2O_2$/Aqua regia | YES |
| T105-075 | — | — | — |
| T105-076 | N/A | N/A | N/A |
| T105-077 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-078 | Wet digestion | $HNO_3$ | YES |
| T105-079 | Wet digestion | $HNO_3/HCl$ | NO |
| T105-080 | Microwave-assisted digestion | $HNO_3$ | --- |
| T105-081 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-082 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-083 | Dry- Ash Digestion- AOAC 999.11 | --- | --- |
| T105-084 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-085 | Microwave-assisted digestion | $HNO_3/HCl/H_2O_2$ | NO |
| T105-086 | Dry ashing | $HNO_3/HCl$ | NO |
| T105-087 | Dry ashing | $HNO_3/HCl$ | NO |
| T105-088 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-089 | --- | --- | --- |
| T105-090 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-091 | N/A | N/A | N/A |
| T105-092 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-093 | Wet digestion | $HNO_3/HCl$ | NO |
| T105-094 | Dry ashing | $HNO_3$ | YES |
| T105-095 | Wet digestion | $HNO_3/HF$ | NO |
| T105-096 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-097 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | NO |
| T105-098 | N/A | N/A | N/A |
| T105-099 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-100 | — | — | — |
| T105-101 | Microwave-assisted digestion | $HNO_3/H_2O_2$/Aqua regia | NO |
| T105-102 | Dry ashing | $HNO_3$ | NO |

| Lab Code | Digestion technique | Digestion medium | Matrix separation |
|---|---|---|---|
| T105-103 | N/A | N/A | N/A |
| T105-104 | Dry ashing | HCl | NO |
| T105-105 | N/A | N/A | N/A |
| T105-106 | Microwave-assisted digestion | $HNO_3/HCl/H_2O_2$ | --- |
| T105-107 | Wet digestion | $HNO_3/HCl$ | NO |
| T105-108 | --- | --- | --- |
| T105-109 | Microwave digestion | 5% $HNO_3$ | --- |
| T105-110 | Microwave-assisted digestion | $HNO_3/H_2O_2$ | --- |
| T105-111 | Microwave-assisted digestion | $HNO_3 + H_2O_2$ (7:1) | YES |
| T105-112 | Microwave-assisted digestion | $HNO_3$ | NO |
| T105-113 | Dry ashing | HCl | NO |

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

| Lab Code | Use of internal standard? | Correction for recovery? | Instrumental method | Method validation? |
|---|---|---|---|---|
| T105-001 | YES | NO | ICP-MS | YES |
| T105-002 | NO | YES | Flame AAS | YES |
| T105-003 | NO | YES | Graphite AAS | YES |
| T105-004 | N/A | N/A | N/A | N/A |
| T105-005 | — | — | — | — |
| T105-006 | NO | NO | ICP-AES | YES |
| T105-007 | NO | NO | Flame AAS | YES |
| T105-008 | YES | NO | ICP-AES | YES |
| T105-009 | NO | NO | Flame AAS | YES |
| T105-010 | NO | NO | ICP-OES | YES |
| T105-011 | N/A | N/A | N/A | N/A |
| T105-012 | — | — | — | — |
| T105-013 | NO | NO | ICP-OES | NO |
| T105-014 | NO | YES | Flame AAS | YES |
| T105-015 | NO | NO | Flame AAS | NO |
| T105-016 | YES | NO | ICP-OES | YES |
| T105-017 | N/A | N/A | N/A | N/A |
| T105-018 | NO | YES | Flame AAS | NO |
| T105-019 | NO | NO | Flame AAS | YES |
| T105-020 | N/A | N/A | N/A | N/A |
| T105-021 | NO | NO | ICP-AES | YES |
| T105-022 | NO | NO | Graphite AAS | NO |
| T105-023 | — | — | — | — |
| T105-024 | NO | YES | ICP-AES / Flame AAS | YES |
| T105-025 | YES | YES | ICP-AES | YES |
| T105-026 | N/A | N/A | N/A | N/A |
| T105-027 | YES | NO | ICP-MS | YES |
| T105-028 | NO | NO | Flame AAS | NO |
| T105-029 | NO | YES | ICP-AES | NO |
| T105-030 | — | — | — | — |
| T105-031 | NO | YES | MP-AES | YES |
| T105-032 | NO | NO | Flame AAS | YES |

| Lab Code | Use of internal standard? | Correction for recovery? | Instrumental method | Method validation? |
|---|---|---|---|---|
| T105-033 | YES | YES | ICP-MS | YES |
| T105-034 | YES | NO | ICP-AES | YES |
| T105-035 | NO | NO | ICP-AES | --- |
| T105-036 | — | — | — | — |
| T105-037 | YES | NO | ICP-MS | YES |
| T105-038 | --- | NO | Flame AAS | YES |
| T105-039 | YES | NO | ICP-AES | YES |
| T105-040 | N/A | N/A | N/A | N/A |
| T105-041 | NO | NO | ICP-OES | YES |
| T105-042 | NO | NO | ICP-AES | YES |
| T105-043 | — | — | — | — |
| T105-044 | YES | NO | ICP-MS | YES |
| T105-045 | YES | NO | ICP-MS | YES |
| T105-046 | YES | NO | ICP-AES | YES |
| T105-047 | NO | NO | ICP-MS | NO |
| T105-048 | YES | NO | Flame AAS | YES |
| T105-049 | NO | NO | Flame AAS | YES |
| T105-050 | YES | NO | ICP-AES | YES |
| T105-051 | NO | YES | ICP-AES | NO |
| T105-052 | N/A | N/A | N/A | N/A |
| T105-053 | NO | NO | Flame AAS | NO |
| T105-054 | — | — | — | — |
| T105-055 | NO | NO | Flame AAS | YES |
| T105-056 | NO | NO | ICP-MS | NO |
| T105-057 | NO | NO | ICP-AES | YES |
| T105-058 | NO | NO | Flame AAS | YES |
| T105-059 | NO | NO | Flame AAS | YES |
| T105-060 | NO | NO | ICP-AES | YES |
| T105-061 | YES | YES | ICP-AES | NO |
| T105-062 | YES | NO | ICP-MS | YES |
| T105-063 | YES | YES | ICP-AES | YES |
| T105-064 | NO | YES | Flame AAS | YES |
| T105-065 | YES | NO | ICP-AES | YES |
| T105-066 | YES | NO | ICP-AES | YES |

| Lab Code | Use of internal standard? | Correction for recovery? | Instrumental method | Method validation? |
|---|---|---|---|---|
| T105-067 | NO | NO | ICP-OES | YES |
| T105-068 | --- | YES | Flame AAS | YES |
| T105-069 | YES | NO | ICP-MS | YES |
| T105-070 | NO | NO | ICP-AES | YES |
| T105-071 | — | — | — | — |
| T105-072 | N/A | N/A | N/A | N/A |
| T105-073 | YES | NO | ICP-MS | YES |
| T105-074 | --- | --- | Graphite AAS | --- |
| T105-075 | — | — | — | — |
| T105-076 | N/A | N/A | N/A | N/A |
| T105-077 | NO | YES | ICP-OES | NO |
| T105-078 | NO | NO | ICP-AES | YES |
| T105-079 | NO | NO | Flame AAS | NO |
| T105-080 | YES | NO | ICP-AES | NO |
| T105-081 | NO | NO | ICP-OES | YES |
| T105-082 | YES | NO | ICP-MS | NO |
| T105-083 | --- | --- | ICP-OES | --- |
| T105-084 | NO | NO | ICP-AES | --- |
| T105-085 | NO | NO | ICP-AES | YES |
| T105-086 | NO | NO | Flame AAS | YES |
| T105-087 | NO | NO | Flame AAS | NO |
| T105-088 | NO | NO | ICP-MS | YES |
| T105-089 | NO | NO | ICP-MS | YES |
| T105-090 | YES | NO | ICP-MS | YES |
| T105-091 | N/A | N/A | N/A | N/A |
| T105-092 | NO | YES | ICP-OES | YES |
| T105-093 | YES | NO | ICP-MS | YES |
| T105-094 | NO | NO | ICP-AES | YES |
| T105-095 | YES | NO | ICP-OES | NO |
| T105-096 | YES | NO | ICP-MS | YES |
| T105-097 | YES | NO | Flame AAS | YES |
| T105-098 | N/A | N/A | N/A | N/A |
| T105-099 | YES | NO | ICP-OES | YES |

| Lab Code | Use of internal standard? | Correction for recovery? | Instrumental method | Method validation? |
|---|---|---|---|---|
| T105-100 | — | — | — | — |
| T105-101 | NO | YES | Flame AAS | YES |
| T105-102 | NO | NO | Flame AAS | YES |
| T105-103 | N/A | N/A | N/A | N/A |
| T105-104 | YES | NO | ICP-AES | YES |
| T105-105 | N/A | N/A | N/A | N/A |
| T105-106 | NO | NO | ICP-MS | NO |
| T105-107 | YES | NO | ICP-MS | NO |
| T105-108 | NO | NO | Spectrophotometer | NO |
| T105-109 | YES | NO | ICP-MS | NO |
| T105-110 | NO | NO | Flame AAS | YES |
| T105-111 | NO | YES | Flame AAS | YES |
| T105-112 | NO | NO | ICP-AES | YES |
| T105-113 | NO | NO | ICP-AES | YES |

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

| Lab Code | Use of internal standard? | Correction for recovery? | Instrumental method | Method validation? |
|---|---|---|---|---|
| T105-001 | YES | NO | ICP-MS | YES |
| T105-002 | NO | YES | Flame AAS | YES |
| T105-003 | NO | YES | Graphite AAS | YES |
| T105-004 | N/A | N/A | N/A | N/A |
| T105-005 | — | — | — | — |
| T105-006 | YES | NO | ICP-MS | YES |
| T105-007 | NO | NO | Flame AAS | YES |
| T105-008 | YES | NO | ICP-AES | YES |
| T105-009 | NO | NO | Flame AAS | YES |
| T105-010 | NO | NO | ICP-OES | YES |
| T105-011 | N/A | N/A | N/A | N/A |
| T105-012 | — | — | — | — |
| T105-013 | NO | NO | ICP-OES | NO |
| T105-014 | NO | YES | Flame AAS | YES |
| T105-015 | NO | NO | Flame AAS | NO |
| T105-016 | YES | NO | ICP-OES | YES |
| T105-017 | N/A | N/A | N/A | N/A |
| T105-018 | NO | NO | Flame AAS | YES |
| T105-019 | NO | NO | Flame AAS | YES |
| T105-020 | N/A | N/A | N/A | N/A |
| T105-021 | NO | NO | ICP-AES | YES |
| T105-022 | NO | NO | Flame AAS | NO |
| T105-023 | — | — | — | — |
| T105-024 | NO | YES | ICP-AES/Flame AAS | YES |
| T105-025 | YES | YES | ICP-AES | YES |
| T105-026 | N/A | N/A | N/A | N/A |
| T105-027 | YES | NO | ICP-MS | YES |
| T105-028 | NO | NO | Flame AAS | NO |
| T105-029 | NO | YES | ICP-AES | NO |
| T105-030 | — | — | — | — |
| T105-031 | NO | YES | MP-AES | YES |
| T105-032 | NO | NO | Flame AAS | YES |

| Lab Code | Use of internal standard? | Correction for recovery? | Instrumental method | Method validation? |
|---|---|---|---|---|
| T105-033 | YES | YES | ICP-MS | YES |
| T105-034 | YES | NO | ICP-AES | YES |
| T105-035 | NO | NO | ICP-AES | --- |
| T105-036 | — | — | — | — |
| T105-037 | YES | NO | ICP-MS | YES |
| T105-038 | --- | NO | Flame AAS | YES |
| T105-039 | YES | NO | ICP-AES | YES |
| T105-040 | N/A | N/A | N/A | N/A |
| T105-041 | NO | NO | ICP-OES | YES |
| T105-042 | NO | NO | ICP-AES | YES |
| T105-043 | — | — | — | — |
| T105-044 | YES | NO | ICP-MS | YES |
| T105-045 | YES | NO | ICP-MS | YES |
| T105-046 | YES | NO | ICP-AES | YES |
| T105-047 | NO | NO | ICP-MS | NO |
| T105-048 | YES | NO | Flame AAS | NO |
| T105-049 | NO | NO | Flame AAS | YES |
| T105-050 | YES | NO | ICP-MS | YES |
| T105-051 | NO | YES | ICP-AES | NO |
| T105-052 | N/A | N/A | N/A | N/A |
| T105-053 | NO | NO | Flame AAS | NO |
| T105-054 | — | — | — | — |
| T105-055 | NO | NO | Flame AAS | YES |
| T105-056 | NO | NO | ICP-MS | NO |
| T105-057 | NO | NO | ICP-AES | YES |
| T105-058 | NO | NO | Flame AAS | YES |
| T105-059 | NO | NO | Flame AAS | YES |
| T105-060 | NO | NO | ICP-AES | YES |
| T105-061 | YES | YES | ICP-AES | NO |
| T105-062 | YES | NO | ICP-MS | YES |
| T105-063 | YES | YES | ICP-AES | YES |
| T105-064 | NO | YES | Flame AAS | YES |
| T105-065 | YES | NO | ICP-AES | YES |
| T105-066 | YES | NO | ICP-AES | YES |

| Lab Code | Use of internal standard? | Correction for recovery? | Instrumental method | Method validation? |
|---|---|---|---|---|
| T105-067 | NO | NO | ICP-OES | YES |
| T105-068 | NO | YES | Flame AAS | YES |
| T105-069 | YES | NO | ICP-AES | YES |
| T105-070 | NO | NO | ICP-AES | YES |
| T105-071 | — | — | — | — |
| T105-072 | N/A | N/A | N/A | N/A |
| T105-073 | YES | NO | ICP-MS | YES |
| T105-074 | --- | --- | Graphite AAS | --- |
| T105-075 | — | — | — | — |
| T105-076 | N/A | N/A | N/A | N/A |
| T105-077 | NO | YES | ICP-OES | NO |
| T105-078 | NO | NO | ICP-AES | YES |
| T105-079 | NO | NO | Flame AAS | NO |
| T105-080 | YES | NO | ICP-AES | NO |
| T105-081 | NO | NO | ICP-OES | YES |
| T105-082 | YES | NO | ICP-MS | NO |
| T105-083 | --- | --- | ICP-OES | --- |
| T105-084 | NO | NO | ICP-AES | --- |
| T105-085 | NO | NO | ICP-AES | YES |
| T105-086 | NO | NO | Flame AAS | YES |
| T105-087 | NO | NO | Flame AAS | NO |
| T105-088 | NO | NO | ICP-MS | YES |
| T105-089 | --- | --- | --- | --- |
| T105-090 | YES | NO | ICP-MS | YES |
| T105-091 | N/A | N/A | N/A | N/A |
| T105-092 | NO | YES | ICP-OES | YES |
| T105-093 | YES | NO | ICP-MS | YES |
| T105-094 | NO | NO | ICP-AES | YES |
| T105-095 | YES | NO | ICP-OES | NO |
| T105-096 | YES | NO | ICP-MS | YES |
| T105-097 | YES | NO | Flame AAS | YES |
| T105-098 | N/A | N/A | N/A | N/A |
| T105-099 | YES | NO | ICP-OES | YES |

| Lab Code | Use of internal standard? | Correction for recovery? | Instrumental method | Method validation? |
|---|---|---|---|---|
| T105-100 | — | — | — | — |
| T105-101 | NO | YES | Flame AAS | YES |
| T105-102 | NO | NO | Flame AAS | YES |
| T105-103 | N/A | N/A | N/A | N/A |
| T105-104 | YES | NO | ICP-AES | YES |
| T105-105 | N/A | N/A | N/A | N/A |
| T105-106 | NO | NO | ICP-MS | NO |
| T105-107 | YES | NO | ICP-MS | YES |
| T105-108 | --- | --- | --- | --- |
| T105-109 | YES | NO | ICP-MS | NO |
| T105-110 | NO | NO | Flame AAS | YES |
| T105-111 | NO | YES | Flame AAS | YES |
| T105-112 | NO | NO | ICP-AES | YES |
| T105-113 | NO | NO | ICP-AES | YES |

**Figure I: Plot of *z*-scores of participants on iron results**

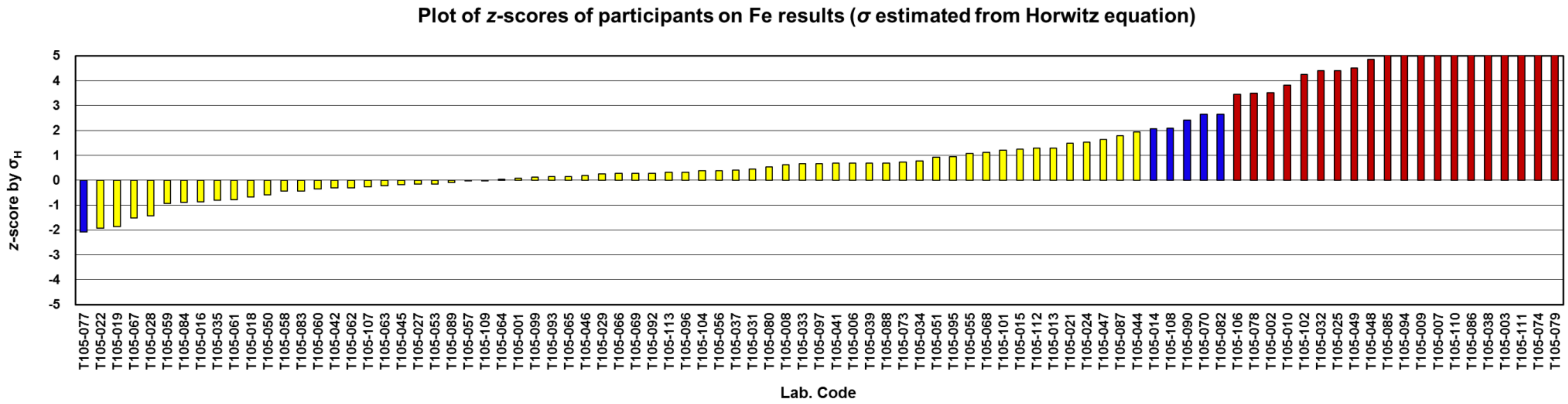


**Figure II: Plot of *z*-scores of participants on zinc results**

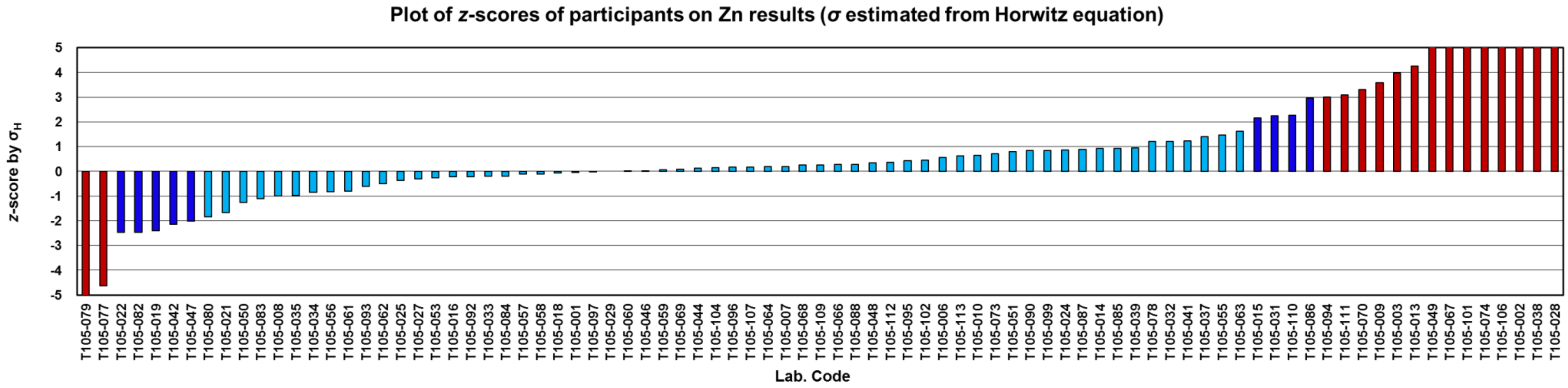


**Figure III: Plot of zeta-scores of participants on iron results**

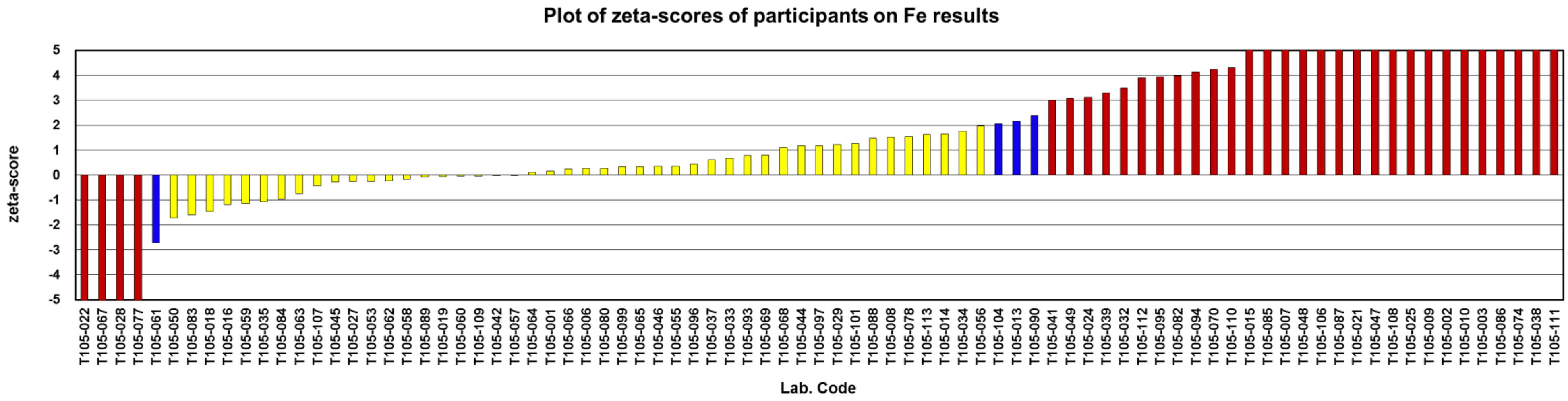
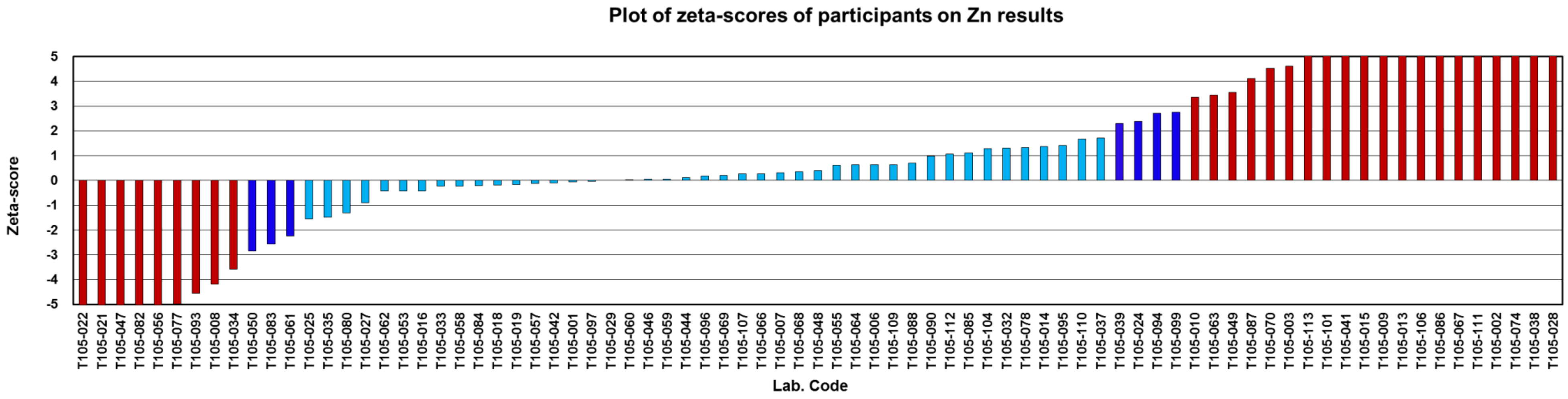
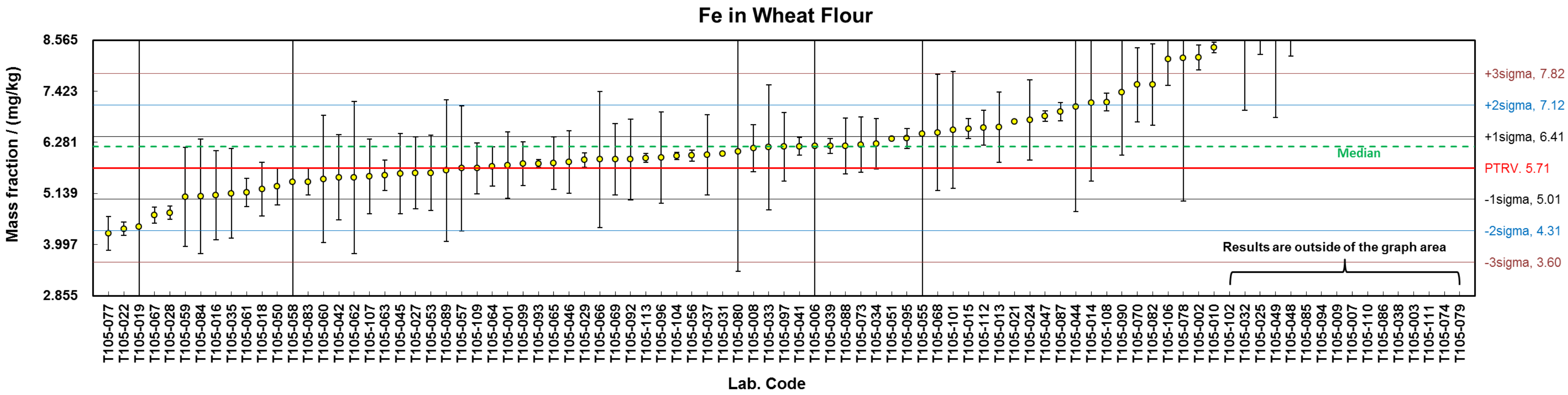


**Figure IV: Plot of zeta-scores of participants on zinc results**



## Figure V: PTRV for iron and participants results



## Figure VI: PTRV for zinc and participants results

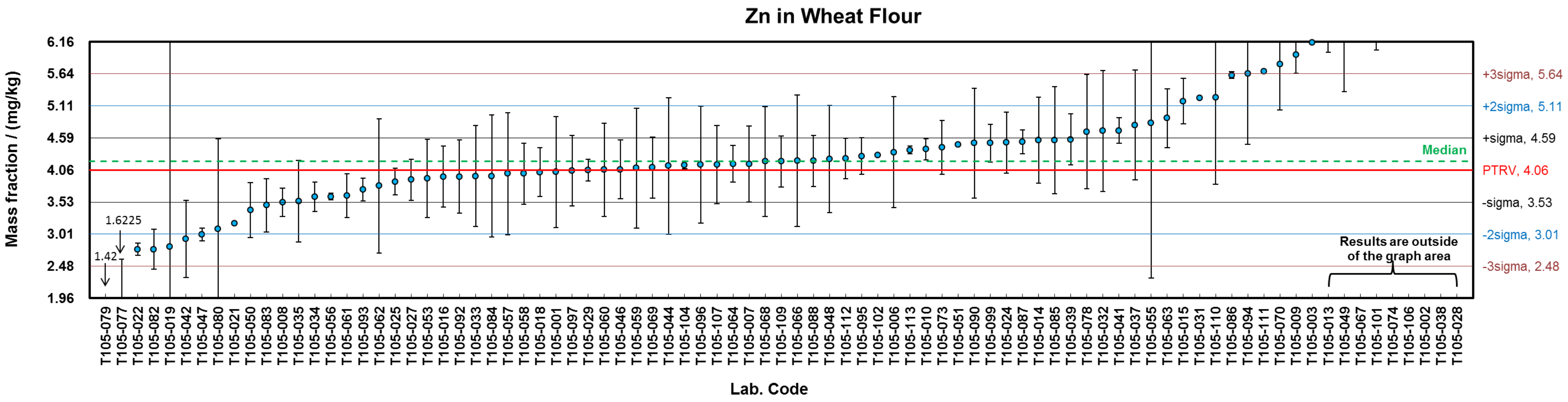
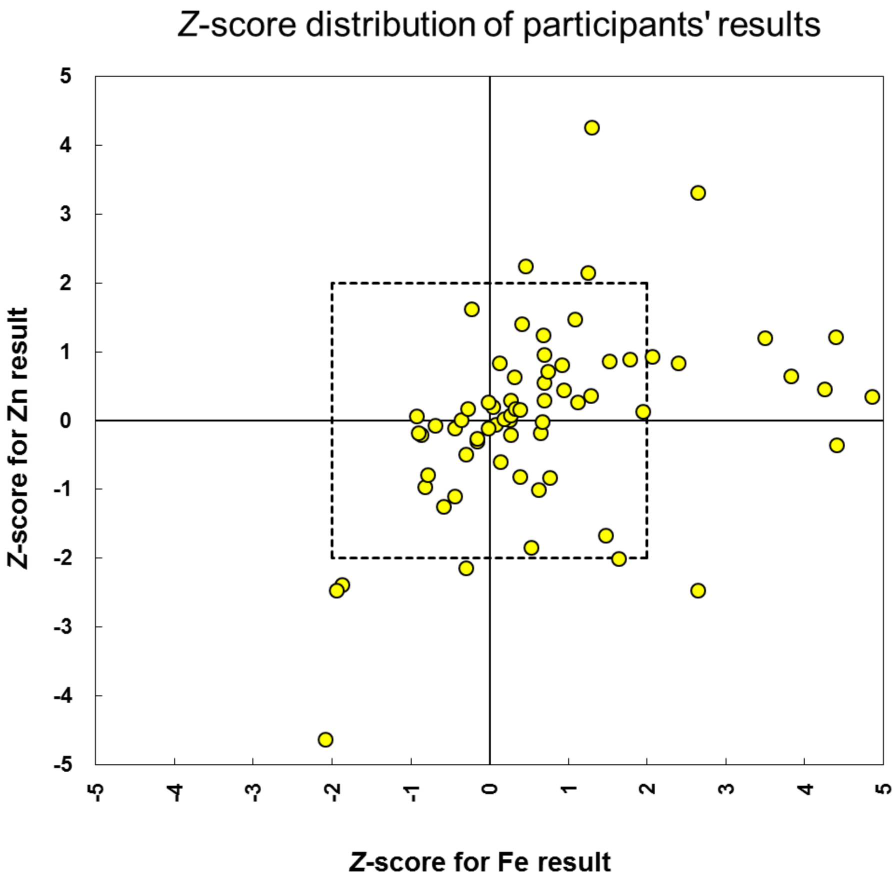


**Figure VII: Youden plot showing z-score and zeta-score distribution of participants' results**



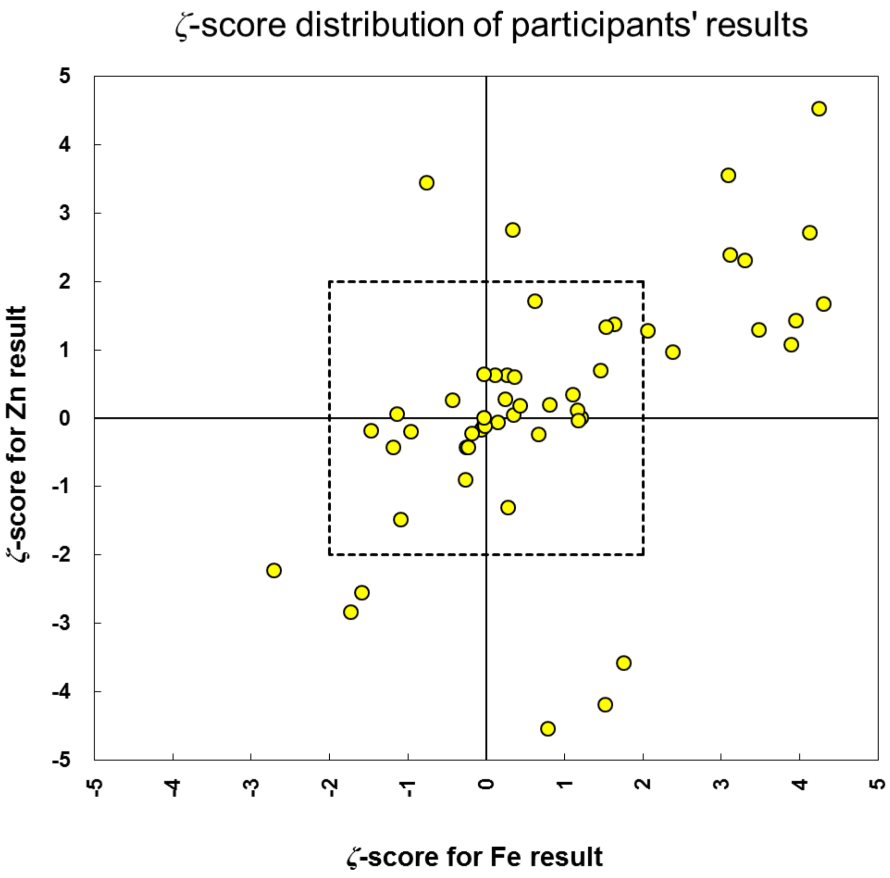



**Figure VIII: Youden plot showing distribution of participants' results with respect to accreditation for the test**

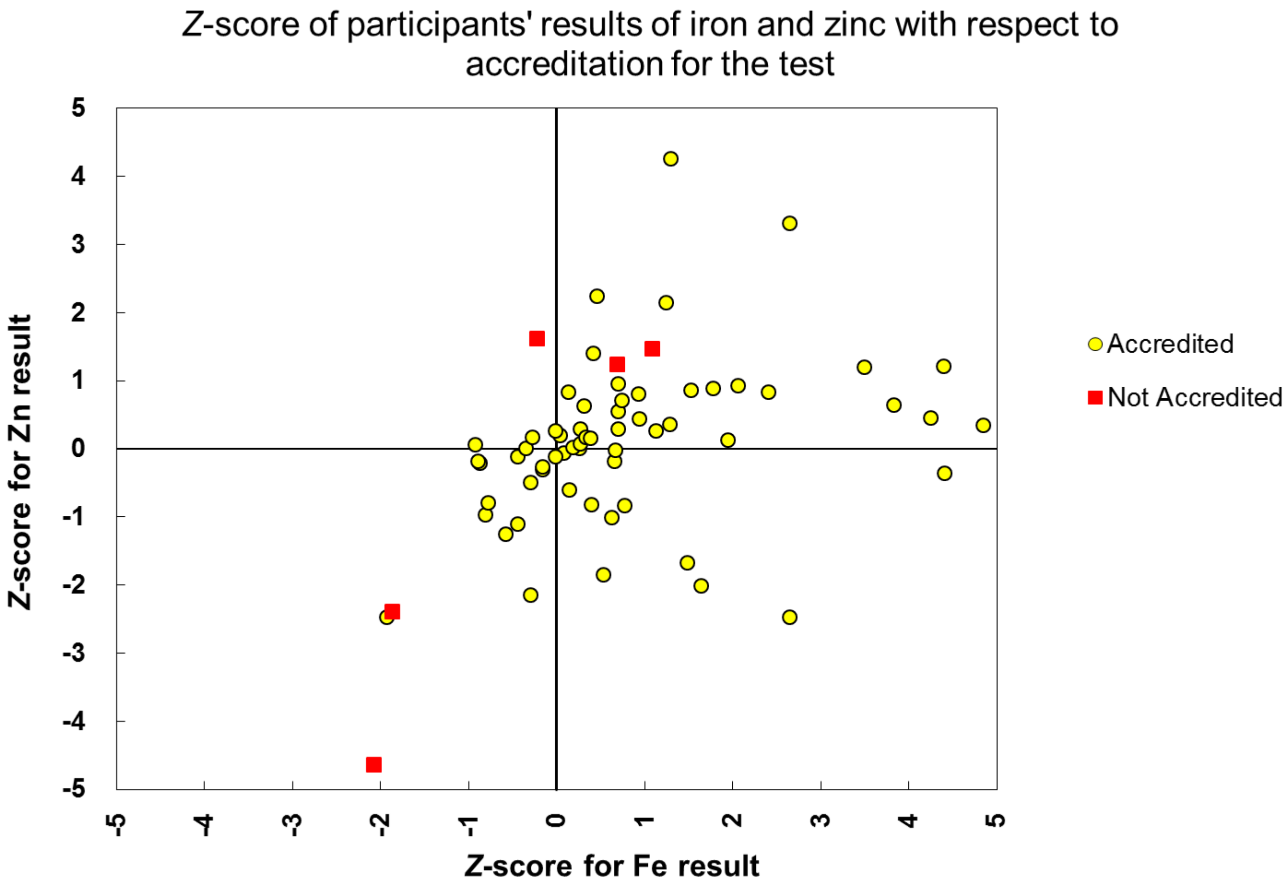


**Figure IX: Youden plot showing distribution of participants' results with respect to their instrumental methods**

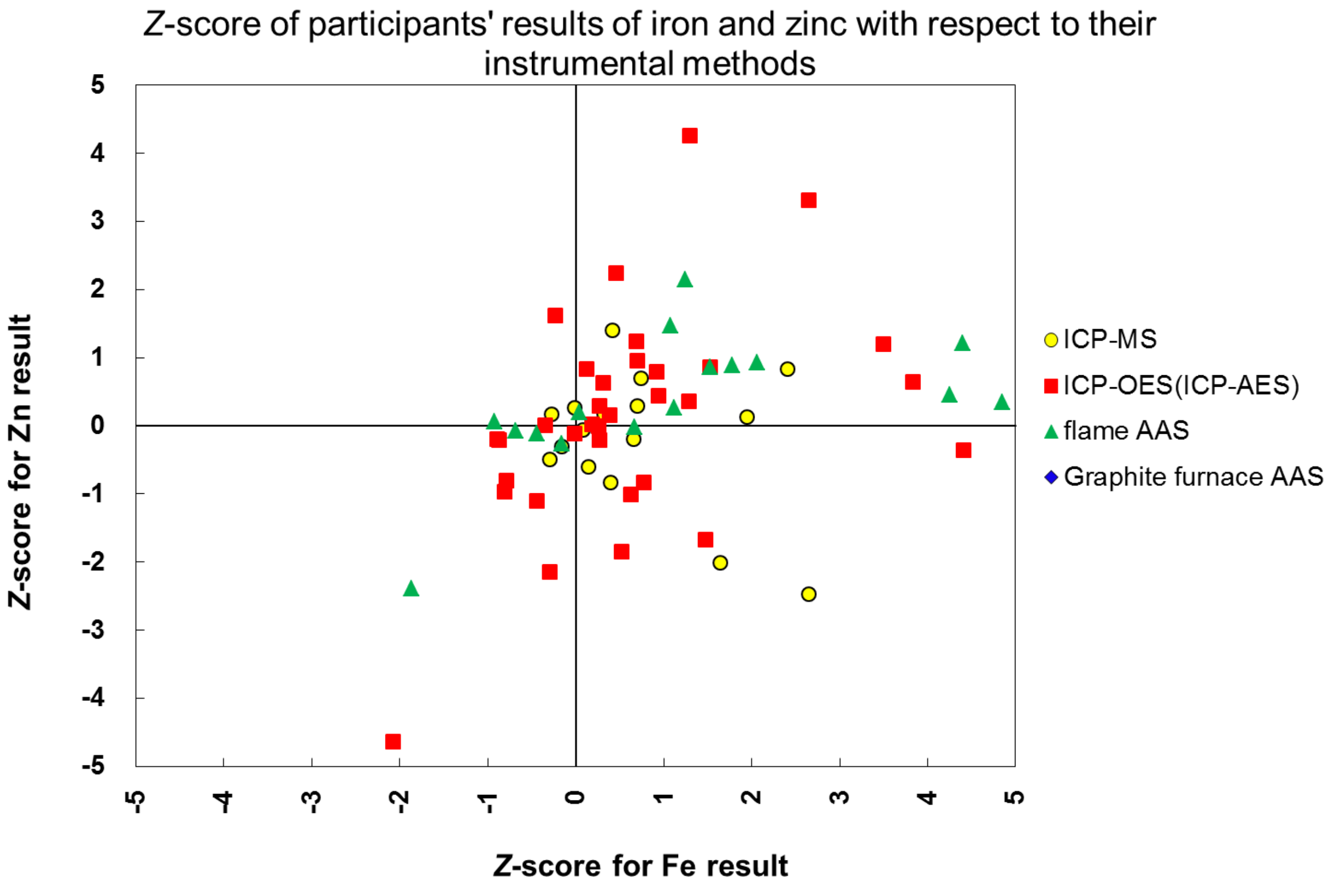
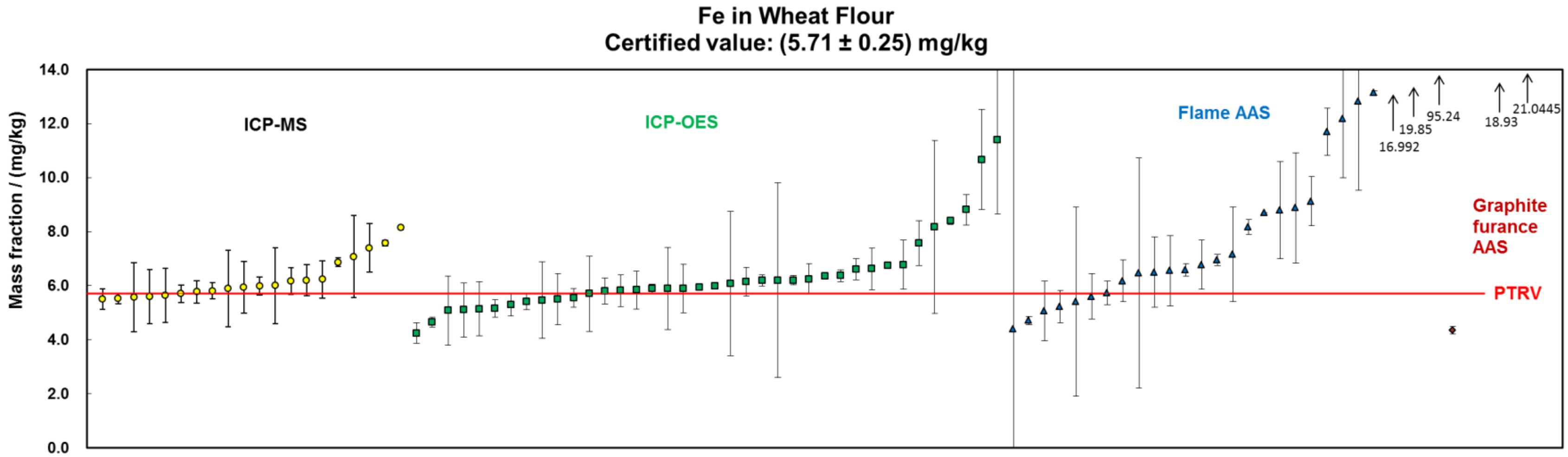


**Figure X: Results from all participants according to participants' instrumental methods**



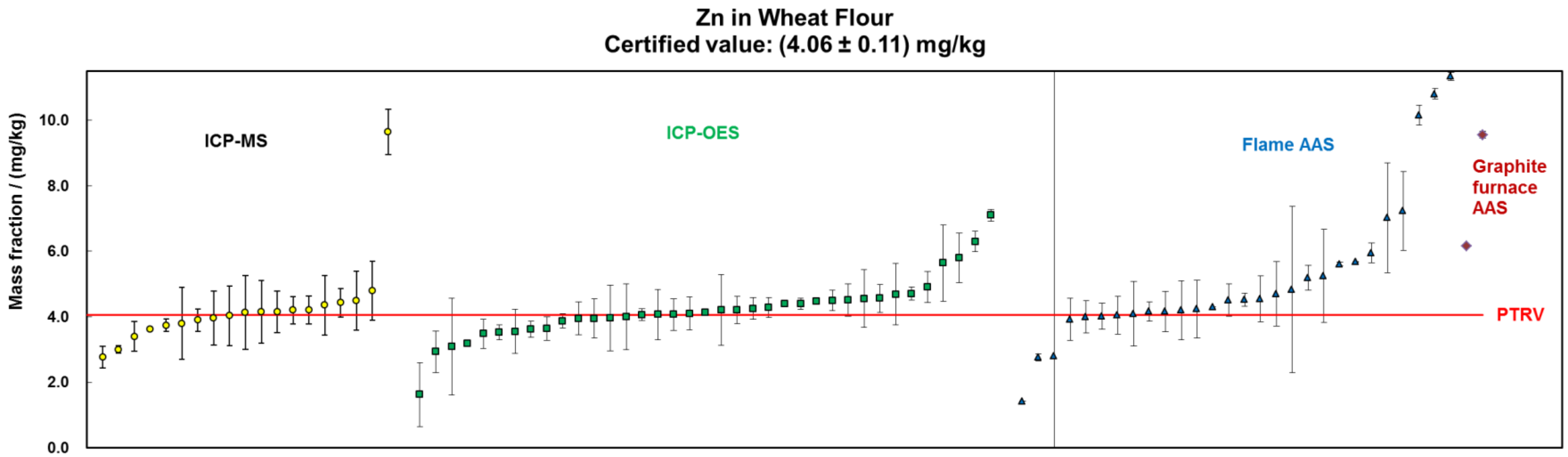



**Figure XI: Youden plot showing distribution of participants' results with respect to the use of internal standard**

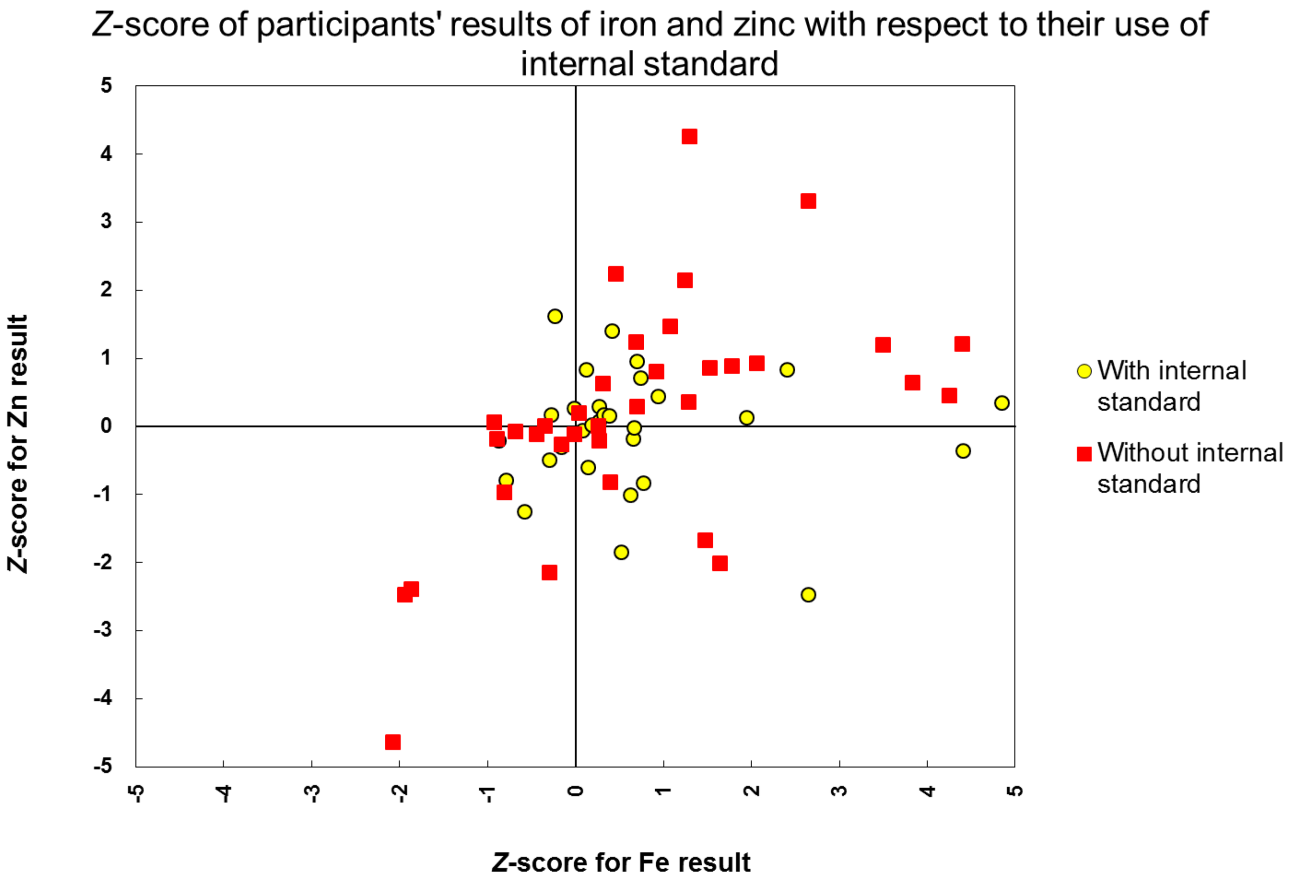
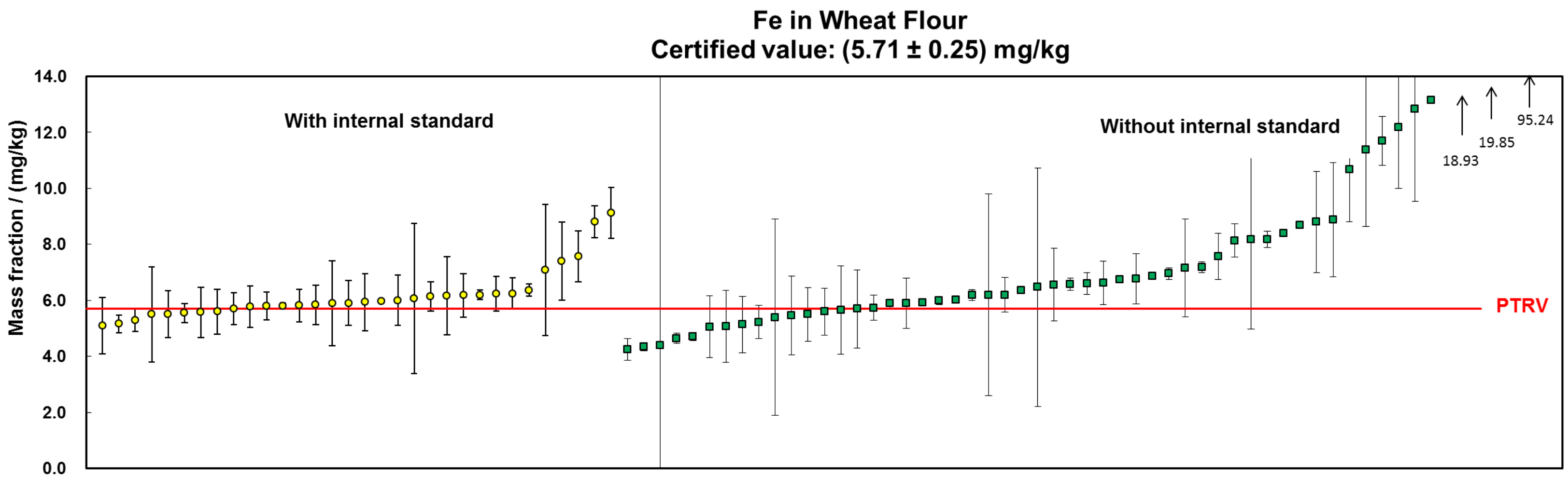


**Figure XII: Results from all participants according to the use of internal standard**



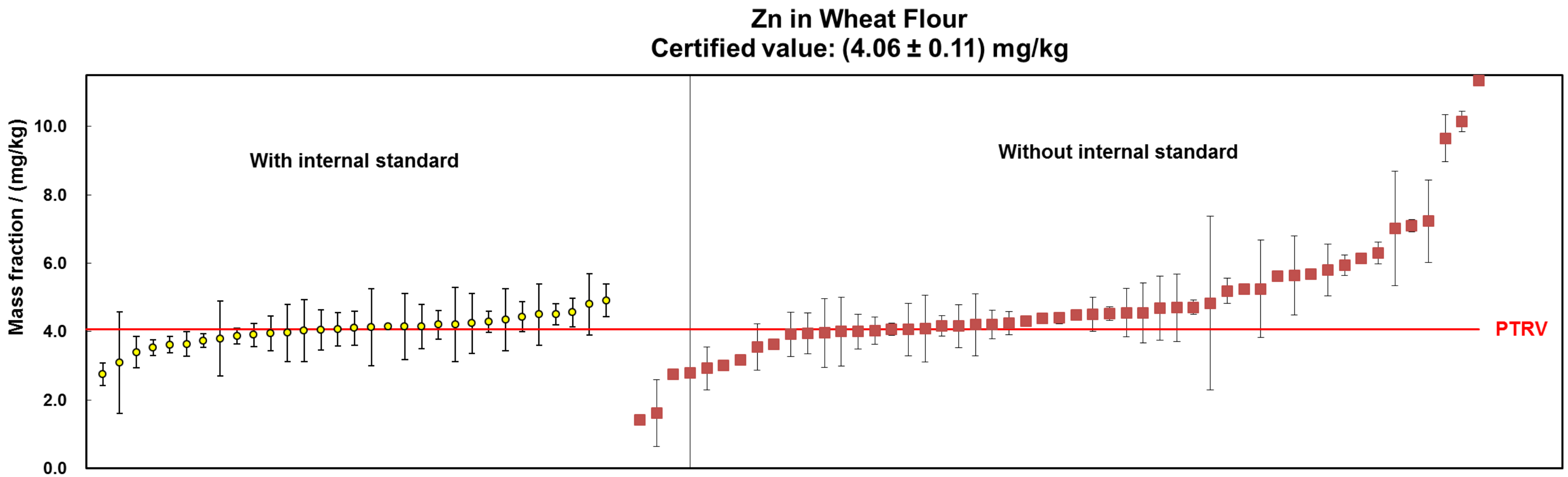



**Figure XIII: Youden plot showing distribution of participants' results with respect to their digestion technique**

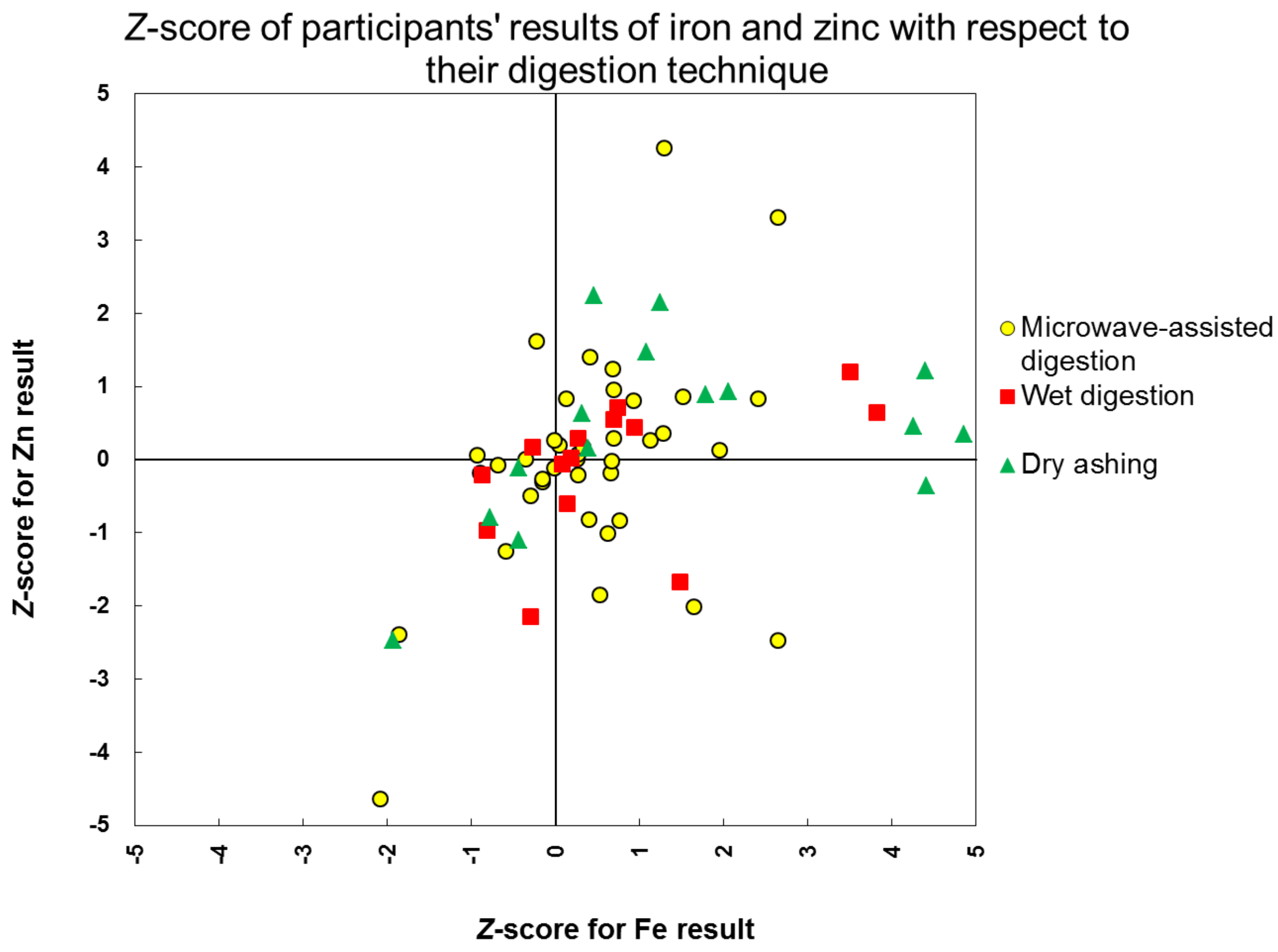
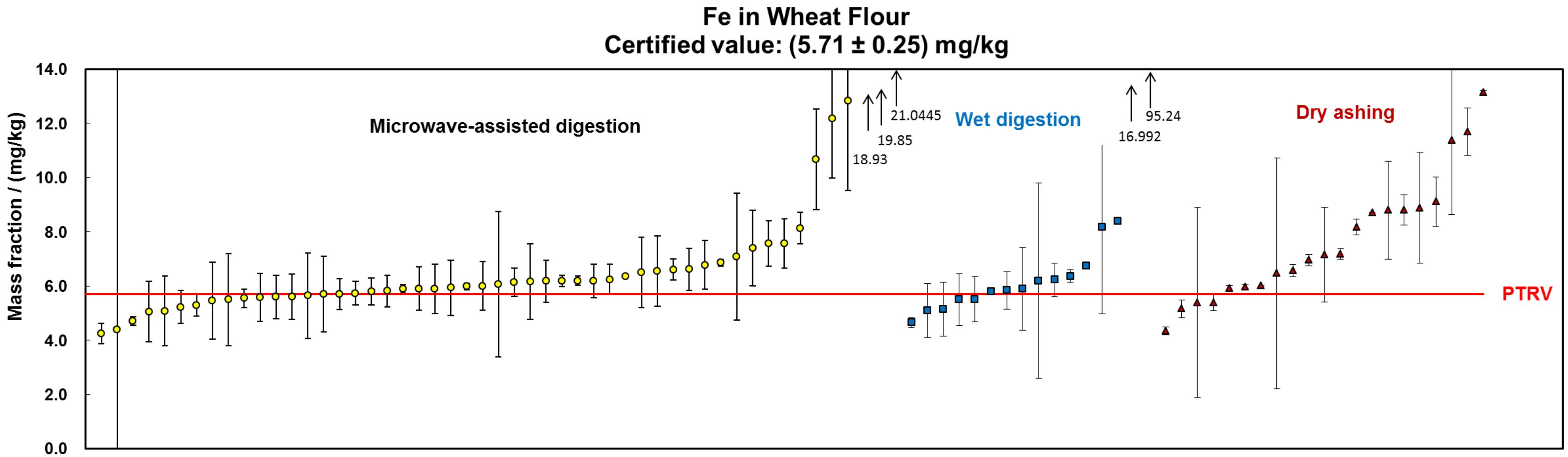


**Figure XIV: Results from all participants according to their digestion technique**



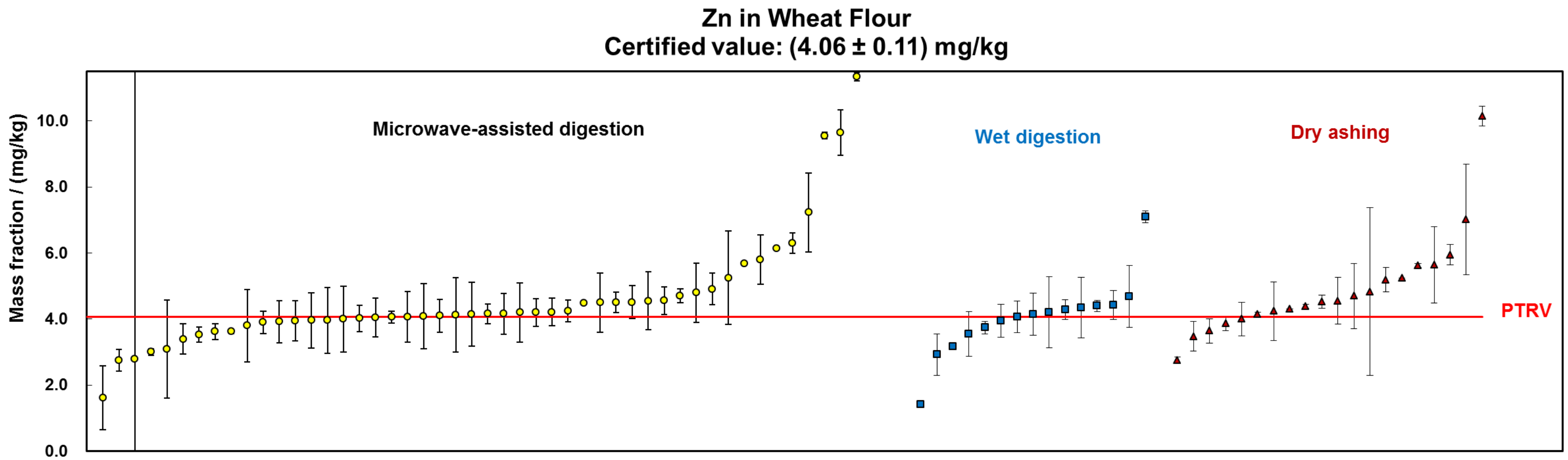



**Figure XV: Youden plot showing distribution of participants' results with respect to the application of recovery correction**

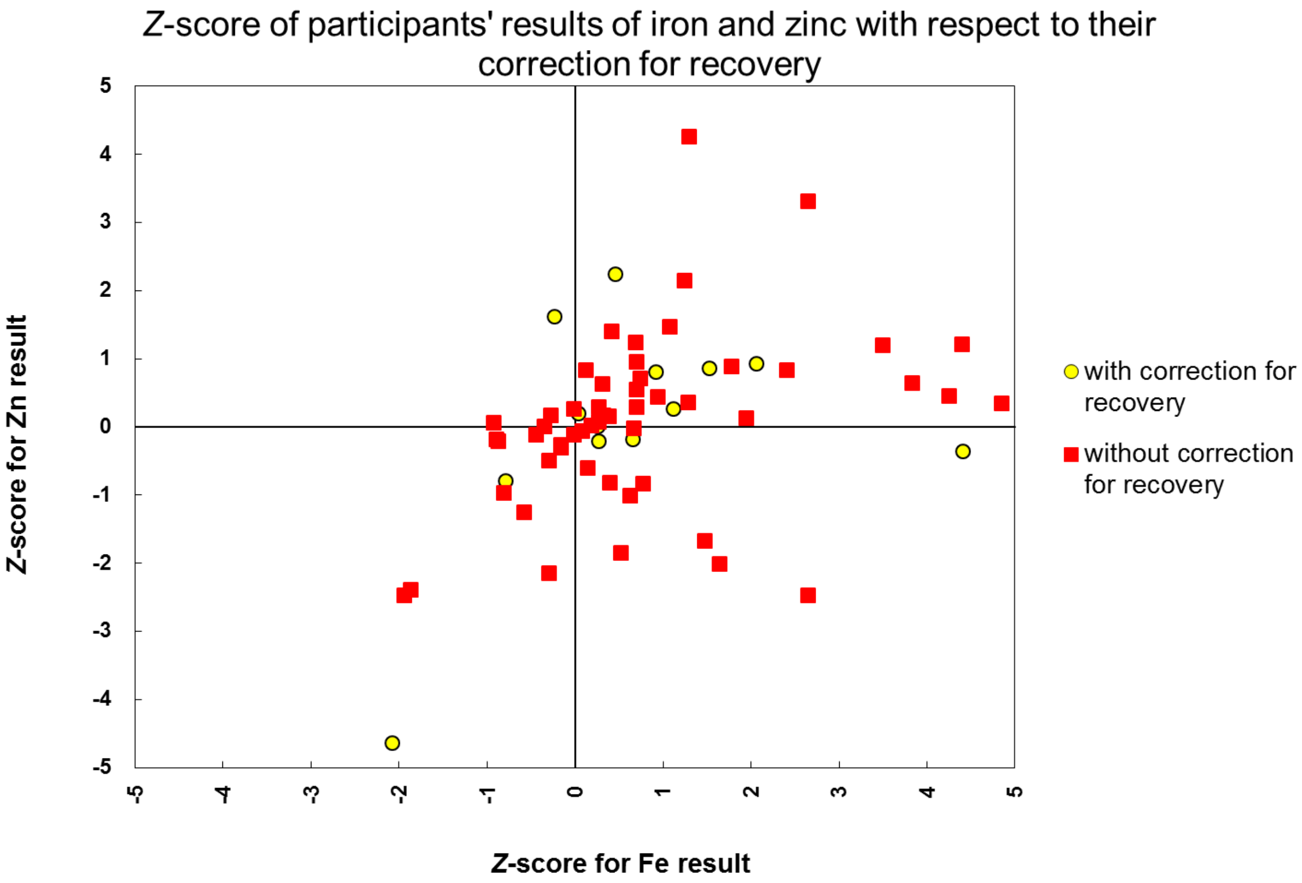
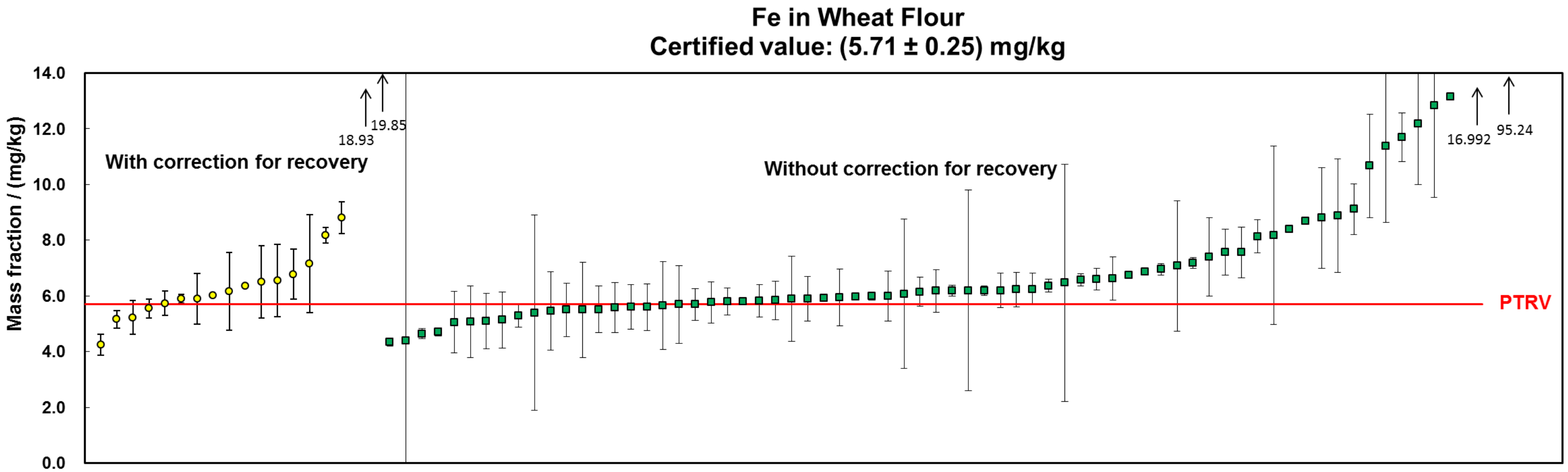


**Figure XVI: Results from all participants according to the application of recovery correction**



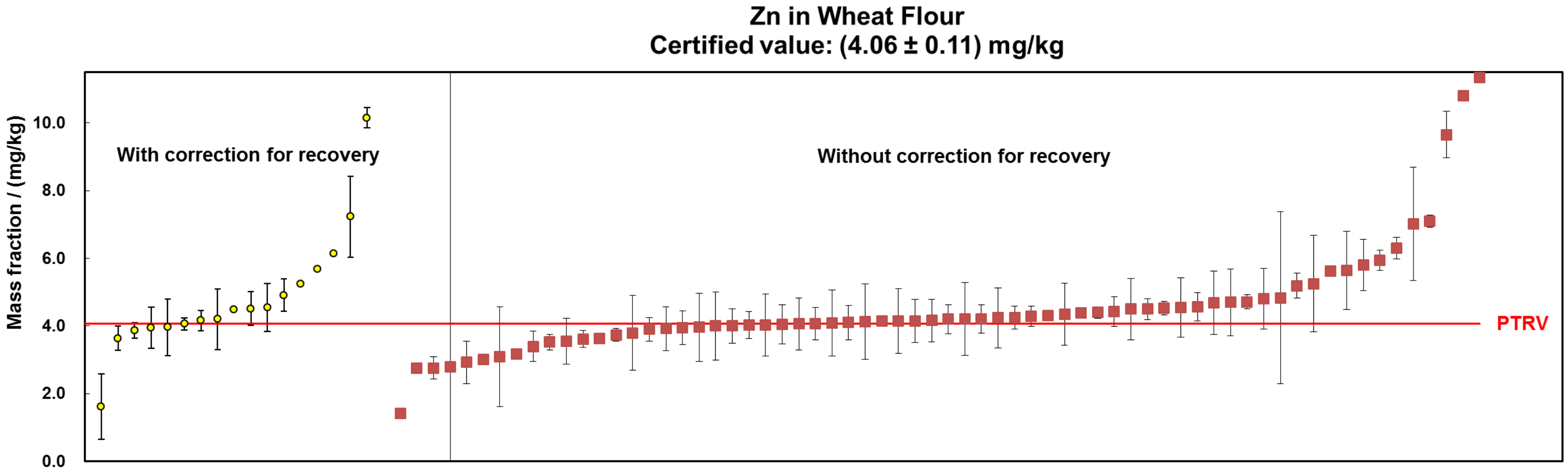



**Figure XVII: Youden plot showing distribution of participants' results with respect to their method validation**

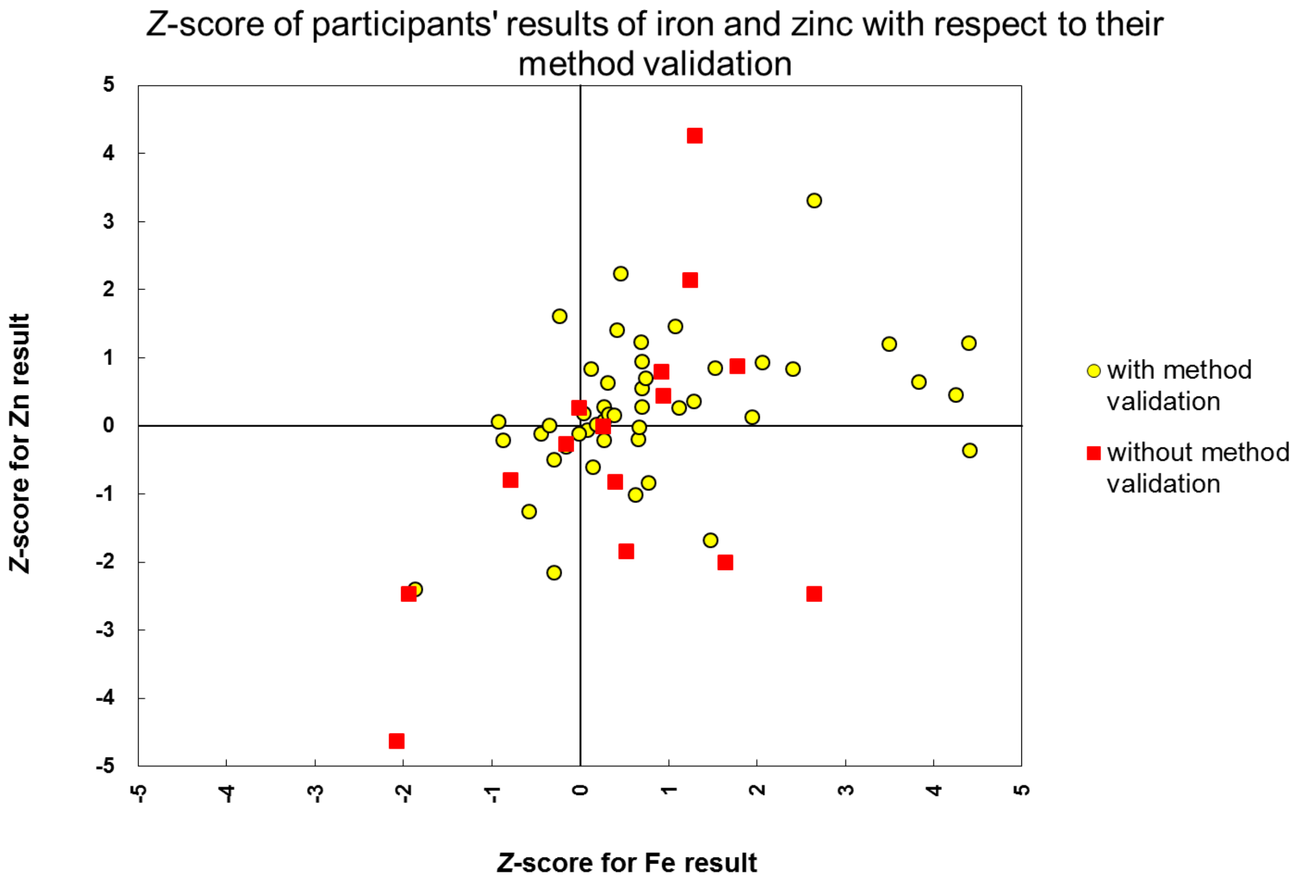
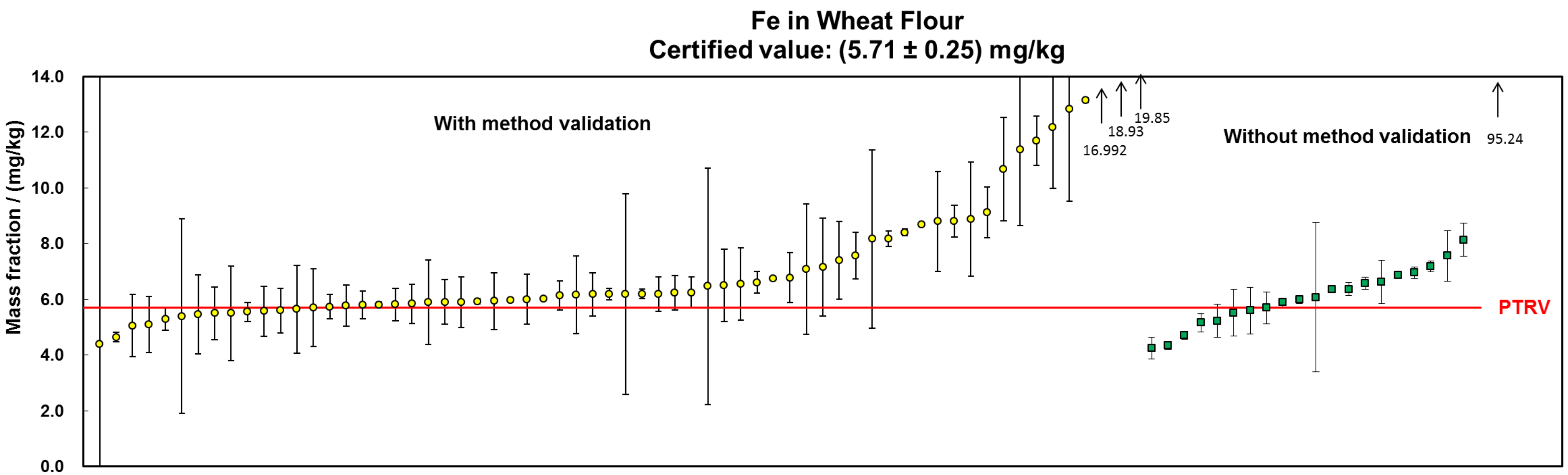
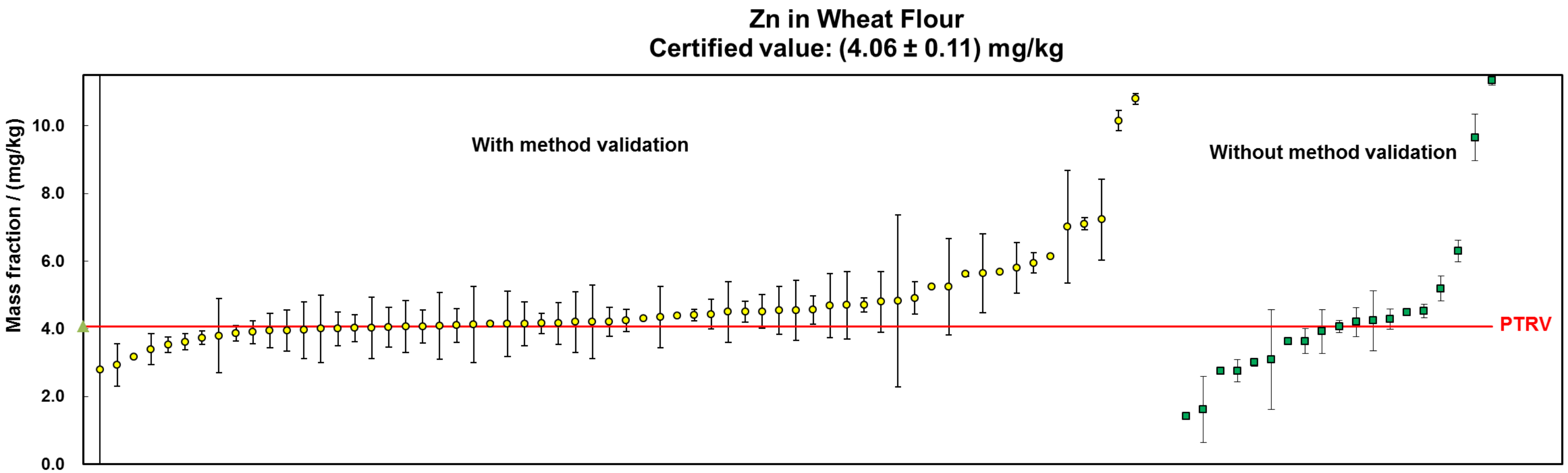


**Figure XVIII: Results from all participants according to their method validation**



**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 ) |
|---|---|
| Iron (Fe) | 1 mg/kg - 20 mg/kg |
| Zinc (Zn) | 1 mg/kg - 20 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 11 and 12 bottles were 1.71 % and 0.31 % for Fe and Zn, 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

- 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.

## 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 standard uncertainty and 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 iron and zinc; and
- Participants should provide information on the methods of analysis (digestion technique and medium, calibration method, use of internal standard, analytical instrument used, correction for recovery, and method validation etc.).

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, **21 October 2016**. 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 (KRISS)
267 Gajeong-Ro, Yuseong-Gu, Daejeon 305-340, Republic of Korea
Tel: +82-42-868-5756
Fax: +82-42-868-5801

## APPENDIX II: Sample Receipt Form

**Sample Receipt Form**
**(Participating Laboratories)**

Laboratory code: ______________________________

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.

## APPENDIX III: Result Report Form

**Result Report**

Laboratory code: ______________________________

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) |
|---|---|---|---|---|
| Iron (Fe) | | | | |
| Zinc (Zn) | | | | |

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

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

| number | Fe (mg/kg) | Zn (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: Iron

a) Sample size used for analysis : ____________________ g ____________________

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

c) Digestion medium: $HNO_3$ / HCl / HF / $H_2SO_4$ / $HClO_4$ / $H_2O_2$ / 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

| | |
|---|---|
| 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: Zinc**

| | |
|---|---|
| a) Sample size used for analysis: | g |
| b) Digestion technique: | Microwave-assisted digestion / Wet digestion / Dry ashing<br>Others (please specify): |
| c) Digestion medium: | $HNO_3$ / HCl / HF / $H_2SO_4$ / $HClO_4$ / $H_2O_2$ / Aqua regia<br>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<br>Others (please specify): |
| i) Correction for recovery: | YES (please specify recovery (%)): / NO |

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

k) Additional information: ______________________

Print name of responsible person:

Date: ____ / ____ /_____.
dd mm yyyy