Asia Pacific Metrology Program (APMP) - Asia Pacific Laboratory Accreditation Cooperation (APLAC) Joint Proficiency Testing Program <Study No. T105>

Nutritional Elements (Iron and Zinc) in Wheat Flour

Proposal

Jointly coordinated by:

Korea Research Institute of Standards and Science (KRISS)

and

Korea Laboratory Accreditation Scheme (KOLAS)

June 2016

APMP-APLAC Joint Proficiency Testing Programme: Nutritional Elements (Iron and Zinc) in Wheat Flour

1. Introduction

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 PT (APLAC PT T100) of toxic elements (lead and cadmium) in wheat flour was conducted in 2015. 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.

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 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 will be conducted in parallel with the APMP-APLAC Joint PT.

Reference values provided by KRISS for iron and zinc 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 National Metrology Institutes (NMIs) with appropriate CMCs as PT reference values for this APMP-APLAC Joint PT 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.

2. Objectives

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

3. Organisers of the joint PT Programme

KRISS (Address: 267 Gajeong-Ro, Yuseong-Gu, Daejeon 305-340, Republic of Korea) is the National Metrology Institute (NMI) of Korea. KRISS takes responsibility for all tasks in the development and operation of the proficiency testing programme, including preparation and distribution of proficiency test samples, data analysis and evaluation of results, preparation and circulation of interim and final reports, and communications with participants.

KOLAS (Address: 93 Isu-Ro, Eumseong-Gun, Chungcheongbuk-Do, Republic of Korea) is the governmental accreditation body of Korea. KOLAS is responsible for proposing the proficiency testing programme and inviting participants and acting as a contact point between APLAC and KRISS.

4. Fee for participation

Free of charge.

5. Call for participation

APLAC members as well as non APLAC members will be invited to participate in the programme. Invitations will be sent to all APLAC members and other accreditation bodies. Participating accreditation bodies will be asked to nominate laboratories to participate and indicate the accreditation status of the nominated laboratories for the test. The number of laboratories shall be limited to 100 due to the limitation of the test sample prepared in KRISS. APLAC members are invited to nominate up to a maximum of 4 laboratories/member body. Non APLAC members are invited to nominate up to a maximum of 2 laboratories/member body. When enrolment exceeds the limit, the number of participating laboratories from each accreditation body will be limited upon discussion with APLAC. Domestic laboratories in Korea can be nominated with no limitation on the number of participants.

Participation will be confirmed within one week after the deadline of the nomination with the assigned lab codes.

6. Test sample

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.

Analytes to be determined 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

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, which are satisfactory for this proficiency testing according to ISO 13528:2015 [1].

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 more than 5 years at room temperature. The stability assessment on similar batches of rice powder CRM and cabbage CRM also demonstrated excellent stability of non-volatile elements in dried food materials. Therefore, the stability of the proficiency test sample is expected to be satisfactory for the purpose of this proficiency testing based on the previous stability studies conducted by KRISS according to the section 6.1.1 in ISO 13528:2015 [1].

7. Reporting and submission of results

Participants should complete the Result Report form which will be distributed to participants. The guideline for reporting results is 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. 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. Under no circumstances will correction or adjustment of analytical data be accepted after the issue of the interim report.

8. Measurement uncertainty

Measurement uncertainty is best estimated within the individual laboratory environment. An estimate of measurement uncertainty is normally based on the combination of a number of influencing parameters (components of uncertainty). As stipulated in ISO Guide to the Expression of Uncertainty in Measurement [2], the uncertainty of each individual parameter should be quantified and expressed numerically as a standard uncertainty. These values are then combined according to the rules of the propagation of uncertainty and the combined standard uncertainty is multiplied by a coverage factor to produce an expanded uncertainty at the 95 % level of confidence.

The standard uncertainty reported from each participant will be used to derive a zeta-score (ζ-score) for evaluation of performance of participants. The expanded uncertainty will be used for calculation of an E_n score just for information.

9. Evaluation of performance of participants

Performance of the participating laboratories will be assessed using *z*-score and ζ-score, which is calculated based on Equations (1) and (2), respectively, as follows:

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

where x_i = the reported result of individual participant
x_{pt} = the assigned value*
σ_{pt} = The standard deviation for proficiency assessment estimated from the Horwitz Equation [3].

* Note: The reference values determined by KRISS will be used as the assigned values. This is in accordance with the ISO/IEC 17043 recommendations on the determination of assigned values for proficiency testing schemes [4].

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

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}

z-Score and ζ-score are commonly interpreted as:

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

Laboratories having a score equal to or larger than 3.0 shall thoroughly investigate their results for the discrepancy and those having a score in the range $2.0 < |z| < 3.0$ are also encouraged to review their results.

10. Issue of reports

An interim report will be issued to participants and their respective accreditation bodies for checking the transcription errors. The draft final report will be then prepared and submitted to APMP-APLAC Joint PT WG for comments and approval. Upon approval, the final report will be reviewed by the APLAC PT Committee for publication. An electronic copy of the final report will be distributed to the participants and their respective accreditation bodies.

11. Proposed program schedule

The proposed time schedule for the various phases of the proficiency testing programme is as follows:

Time schedule	Phase
June 2016	Call for nomination
22 July 2016	Deadline of nomination
5 August 2016	Deadline for registration
22-26 August 2016	Sample dispatch
21 October 2016	Deadline for submission of results
February 2017	Issue of the interim report
June 2017	Issue of the final report

12. Confidentiality

The concerned parties (APMP, APLAC, KOLAS and KRISS) strive to maintain strict confidentiality of the characteristic properties of the proficiency test sample distributed and the performance of all participating laboratories. To preserve the confidentiality, participants receive reports giving all results for assessment but without identifying individual laboratories. The code number assigned to a participant in the proficiency testing programme will be provided only to the contact person/authorized person of the participating laboratory through the respective accreditation body.

13. Contact

For more information on the proficiency testing programme, please contact the coordinator by e-mail (E-mail: aplacpt.inorg@kriss.re.kr).

If you have any query or comment on the proposal, please send it to the following contact points before 30 June 2016:

Dr. Sook Heun Kim or Dr. Euijin Hwang, aplacpt.inorg@kriss.re.kr

14. References

1) ISO 13528:2015, Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons, International Standards Organization, 2015, Geneva, Switzerland.
2) ISO/IEC Guide 98-3:2008, Uncertainty of Measurement – Part 3: Guide to the Expression of Uncertainty in Measurement (GUM:1995), 2008, Geneva, Switzerland.
3) Thompson, M; Ellison, S. L. R.; Wood, R; The international harmonized protocol for the proficiency testing of analytical chemistry laboratories, *Anal. Chem*., **1982**, 54: 67A-76A.

4) ISO/IEC 17043:2010. Conformity Assessment – General Requirements for Proficiency Testing, International Standards Organization, 2010, Geneva, Switzerland.