



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



KRISS 한국표준과학연구원
Korea Research Institute of Standards and Science



**Asia Pacific Metrology Program (APMP) -
Asia Pacific Laboratory Accreditation Cooperation
(APLAC)
Joint Proficiency Testing Programme
<APLAC PT T100>**

Toxic Elements (Lead and Cadmium) in Wheat Flour

Proposal

Jointly coordinated by:

Korea Research Institute of Standards and Science (KRISS)

and

Korea Laboratory Accreditation Scheme (KOLAS)

15 May 2015



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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. Wheat contains a number of elements as well as nutrients. The elements such as copper, zinc, iron, nickel and manganese are essential for our biological functions, but high concentrations of such elements are hazardous to our health. Wheat also may contain certain hazardous elements such as lead, cadmium, arsenic and mercury. Agricultural products like wheat are prone to be contaminated with toxic elements from fertilizer, contaminated agricultural soil and water. Many countries set food safety regulations limiting the amount of toxic elements in agricultural products. With increasing international trade of food and agricultural products, traceable measurements of hazardous elements in agricultural products have become one of the essential requirements for ensuring food safety. Codex Alimentarius Commission (CAC) also set maximum levels of contaminants in food and feed to protect public health. In case of lead or cadmium in wheat the maximum level is 0.2 mg/kg [1].

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 (KRIS), a member of APMP, and the Korea Laboratory Accreditation Scheme (KOLAS), a member of APLAC, have jointly proposed a PT scheme for the determination of toxic elements (lead and cadmium) in wheat flour. The purpose of this study is to demonstrate the capability of participating laboratories in measuring toxic elements in the test sample of wheat flour. A domestic PT round for Ministry of Food and Drug Safety (MFDS) in Korea will be conducted in parallel with the APMP-APLAC joint PT.

Reference values certified by KRIS for cadmium and lead in the test sample will be used as the assigned values for evaluating measurement results of participants. The relevant Calibration and Measurement Capabilities (CMCs) of KRIS 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 at this and forthcoming APMP-APLAC Joint PTs will allow the rigorous evaluation of the accuracy of participants' results. It will enhance the quality of the PT programme and also help



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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 lead and cadmium at mg/kg levels in the test sample of wheat flour by various analytical techniques.

3. Organisers of the joint PT Programme

KRIS (Address: 267 Gajeong-Ro, Yuseong-Gu, Daejeon 305-340, Republic of Korea) is the National Metrology Institute (NMI) of Korea. KRIS 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: 98 Gyoyukwon-ro, Gwacheon-Si, Gyonggi-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 KRIS.

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 KRIS. 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 in number of participation.



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The 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 lead and cadmium solutions were added into the wheat flour to make it into a paste form, and then mixed in a Teflon-coated mixing bowl over 4 hours. The mixed wheat flour paste was then frozen for 2 hours and then dried for over 130 hours using a freeze dryer (PVRFD 100R, Ilshin Lab, Korea) with capacity of 100 kg. The weight loss due to freeze-drying was about 13 %. Freeze-dried wheat flour was ground using a laboratory mill (Pulverisette 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)
Cadmium (Cd)	0.05 mg/kg -1 mg/kg
Lead (Pb)	0.05 mg/kg -1 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 Cd and Pb contents due to between-sample inhomogeneity were less than 0.29 % and 0.23 %, respectively, which are satisfactory for the present proficiency testing according to ISO 13528:2005 [2].

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 the similar batch of rice powder CRM also demonstrated excellent stability of non-volatile elements in dried food materials. Therefore, the stability of the proficiency test sample is expected to be satisfactory for the purpose of present proficiency testing based on the previous stability studies of KRIS.



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7. Reporting and submission of results

Participants should complete the Result Report. The guideline of reporting results is as followings:

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

Participants should be aware that any submitted results are considered final and accordingly such results and units should be thoroughly checked before submission. Participants should submit the Result Report electronically to the coordinator of the proficiency testing programme (E-mail: aplact.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 [3], 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 could be used to derive a zeta-score for information.

9. Evaluation of performance of participants

Performance of the participating laboratories will be assessed using z-score, which is calculated as follows:

$$z = \frac{x_i - \bar{x}}{\sigma_{pt}}$$



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where x_i = the reported result of individual participant
 x = the assigned value*
 s_{pt} = The standard deviation for proficiency assessment estimated from the Horwitz Equation [4].

* Note: The reference values determined by KRIS 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 [5].

z-Score is commonly interpreted as:

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

Laboratories having a $|z|$ score equal to or larger than 3.0 shall thoroughly investigate their results for the discrepancy and those having a z-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 PTWG 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
May 2015	Call for Participation
June 2015	Deadline for registration
July 2015	Distribution of samples
4 September 2015	Deadline for submission of results
December 2015	Issue of the interim report
April 2016	Issue of the final report

12. Confidentiality



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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 and/or 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 15 March 2015:

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

14. References

- 1) Codex Standard 193-1995, Codex General Standard for Contaminants and Toxins in Food and Feed, Codex Alimentarius Commission.
- 2) ISO 13528:2005, Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons, International Standards Organization, 2005, Geneva, Switzerland.
- 3) ISO/IEC G98:1995, Guide to the Expression of Uncertainty in Measurement (GUM), International Standards Organization, 1995, Geneva, Switzerland.
- 4) 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.
- 5) ISO/IEC 17043:2010. Conformity Assessment – General Requirements for Proficiency Testing, International Standards Organization, 2010, Geneva, Switzerland.