



APMP-APLAC Joint Proficiency Testing Programme  
(APLAC T110)  
Toxic Metal/Metalloid Species in Powdered Rice



**Instructions for Participants**

**1. Objectives**

With the aim of enhancing the measurement capability and traceability of measurements in countries and 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) <sup>Note 1</sup> has agreed to strengthen cooperation between the two organizations. At the 2013 APMP General Assembly and Related Meetings, APMP and APLAC agreed to establish the APMP-APLAC Joint Proficiency Testing Working Group (PTWG) as a formal infrastructure to provide more proficiency testing (PT) programmes with metrologically traceable reference values for performance evaluation purpose for participating measurement institutes and laboratories in the region. To echo this initiative, the Government Laboratory, Hong Kong (GLHK) proposed a new joint PT study on the determination of metal contaminants in powdered rice for 2019/20. The purpose of the study is to demonstrate the capability of participating laboratories in measuring toxic metal/metalloid contaminants in rice (powdered form), an important staple food for human consumption. Arsenic, inorganic arsenic [total as the sum of arsenic(III) and arsenic(V)] and cadmium are selected as the target analytes in this PT study.

Rice (*Oryza sativa L.*) is one of the top three most produced and consumed crop worldwide, after wheat and coarse grains. It is being the staple for around 50% of the world's population. It is grown widely in South-East Asia, and with more discrete regional distribution in Southern Europe, Southern USA, South America and Africa. Rice contains a number of metals/metalloids include nutrients such as copper, zinc and selenium, but may also contain certain amount of toxic metals/metalloids such as arsenic and cadmium. Those toxic metals/metalloids may present naturally in the earth's crust, but they may also exist as pollutants from mining and over usage of fertilizers contaminating the environment. Arsenic is a metalloid that occurs in inorganic and organic forms, which are found in the environment from both natural sources and human activities. In general, inorganic forms of arsenic are more toxic to humans than organic ones. Arsenic and cadmium are two of the "Ten chemicals of major public health concern" identified by the World Health Organization (WHO). Many countries set food safety regulations limiting the amount of toxic metal contaminants in agricultural products. With increasing international trade of food and agricultural products, traceable measurements of toxic metal contaminants in agricultural products have become one of the essential requirements for ensuring food safety. The use of reliable methods for

*Note 1: The Asia Pacific Accreditation Cooperation (APAC) was established on 1 January 2019 by the amalgamation of two former regional accreditation cooperations – the Asia Pacific Laboratory Accreditation Cooperation (APLAC) and the Pacific Accreditation Cooperation (PAC).*



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measurement of toxic metal contaminants is important in safeguarding the quality of these products and the public health. The Codex Alimentarius Commission (CAC) set maximum levels of contaminants in food and feed to protect public health. [1] In the case of milled rice, the maximum level of inorganic arsenic and cadmium is 0.2 mg/kg and 0.4 mg/kg respectively. For husked rice, the maximum level of inorganic arsenic is 0.35 mg/kg. The European Union also set maximum levels of inorganic arsenic in foodstuffs in 2015. The maximum level of inorganic arsenic is 0.25 mg/kg in husked rice [2].

The APMP-APLAC Joint Proficiency Testing Programme (APLAC T110) is coordinated by GLHK. The aim of this study is to demonstrate the capability of participating laboratories in measuring the mass fractions of the incurred analytes (e.g. arsenic, inorganic arsenic [total as the sum of arsenic(III) and arsenic(V)] and cadmium) at mg/kg levels in dry mass basis in the proficiency test sample of rice (powdered form) by various analytical techniques.

## 2. Analysis of the proficiency test sample

Participating laboratories will be provided with **ONE** bottle containing about **30 g** of rice powder. Upon receipt of the sample, please complete the Sample Receipt Form and return it to the co-ordinator of the proficiency testing programme (E-mail: [aplact.rice@govtlab.gov.hk](mailto:aplact.rice@govtlab.gov.hk)). Replacement of a new bottle of sample will be arranged if the proficiency test sample is identified to be not suitable for analysis.

The proficiency test sample should be stored under room temperature conditions (about 20 °C).

Participants should treat the proficiency test sample in the same manner as the majority of routinely tested samples. Participants should use the test method of their choice, which should be consistent with their routine procedures. The proficiency test sample should be mixed thoroughly before conducting the tests. The analysis should be conducted with a recommended sample size of at least 0.5 g. Participants are requested to perform at least three independent measurements on three separate portions of the sample and to determine the mass fractions of the analytes. All of the three measurands and the range of values to be expected for the proficiency test sample are given as follows:

Measurand	Mass fraction (expected range of values)
Arsenic (total)	0.1 – 1.0 mg/kg
Inorganic arsenic	0.1 – 1.0 mg/kg
Cadmium	0.2 – 1.0 mg/kg



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Participants should also carry out the dry mass correction. For the determination of dry mass correction, a minimum of three separate portions (recommended size to be about 1 g each) of the sample shall be taken and dried at 135 °C for 1.5 hours, then balanced in a desiccator to room temperature. [3] Dry mass correction shall be carried out at the same time as the test sample portions are to be analysed. Dry mass correction shall be carried out at the same time as the test sample portions are to be analysed.

For safety considerations, the proficiency test sample should be handled with care to avoid from contacting with eyes and inhalation of sample powder. Wash the suffered body areas with plenty of water and consult physicians when necessary.

For this proficiency testing programme, return of the untested proficiency test sample is not necessary. There is no fee for participation.

### 3. Reporting and submission of results

Participants should complete the Result Proforma. The instructions on the manner of reporting test results are as follows:

- Units of measurement: Report the mass fractions of the analytes and associated uncertainties in mg/kg;
- Number of significant figures: Report the test results to 3 significant figures;
- Reporting basis: Report the test results on “dry-mass basis”;
- For each analyte, the mean value of at least three independent measurements, the expanded uncertainty of the mean value and the coverage factor (which gives a level of confidence of approximately 95 %) should be reported;
- Participants should provide information on the methods of analysis; and
- For the determination of dry mass correction, a minimum of three separate portions (recommended size to be about 1 g each) of the sample shall be taken and dried at 135 °C for 1.5 hours, then balanced in a desiccator to room temperature. [3] Dry mass correction shall be carried out at the same time as the test sample portions are to be analysed.

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 Proforma electronically to GLHK (E-mail: [aplacpt.rice@govtlab.gov.hk](mailto:aplacpt.rice@govtlab.gov.hk)) on or 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.



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#### 4. Performance evaluation

The performance of the participating laboratories will be assessed using  $z$ -score, which is calculated as follows [4, 5]:

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

where  $x_i$  : the participant's result  
 $x_{pt}$  : the assigned value\*  
 $\sigma_{pt}$  : the standard deviation for proficiency assessment estimated from the Horwitz equation [6]

\* Note: The reference values obtained from GLHK with the support of CMCs claim GLHK042, GLHK061 and GLHK021 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].

$z$ -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.

For reference purpose, the performance of the participating laboratories will be assessed using zeta-score ( $\zeta$ ), which is calculated as follows [5]:

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

where  $x_i$  : the participant's result  
 $x_{pt}$  : the assigned value  
 $u(x_i)$  : the participant's own estimate of the standard uncertainty of its result  $x_i$ .  
 $u(x_{pt})$  : the standard uncertainty of the assigned value  $x_{pt}$



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$\zeta$ -scores are interpreted as in the same way as  $z$ -scores using the same critical values of 2.0 and 3.0.  $\zeta$ -scores may be used in conjunction with  $z$ -scores, as an aid for improving the performance of laboratories as follows. If a laboratory obtains  $|z|$  scores that exceed 3.0, they may find it of value to examine their test procedure step by step and derive an uncertainty budget for that procedure. The uncertainty budget will identify the steps in the procedure where the largest uncertainties arise, so that the laboratory can see where to expend effort to achieve an improvement. If their  $|\zeta|$  scores also exceed the critical value of 3.0, it implies that their uncertainty budget does not include all significant sources of uncertainty [5]. Laboratories are encouraged to review their uncertainty budget.

Upon approval, an electronic copy of the final report on the performance of participating laboratories will be tentatively distributed to the participants. The report will reveal only the code number assigned to the designated participating laboratory and the identity of participants in this proficiency testing programme will be kept confidential. A summary of the final report will be posted on the website of GLHK for public information.

## 5. Programme schedule

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

Time schedule	Phase
February 2019	Call for Participation
31 March 2019	Deadline for registration
April/May 2019	Distribution of samples
30 September 2019	Deadline for submission of results
December 2019	Issue of interim report
June 2020	Issue of final report

## 6. Confidentiality

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.

The concerned parties (APMP, APAC and GLHK) strive to maintain strict confidentiality with respect to composition of the proficiency test samples distributed and the performance of all participating laboratories. To preserve the confidentiality, participants receive report(s) giving all results for assessment but without identifying individual



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laboratories. The code number assigned to a participant in the proficiency testing programme is only made known to the contact person/authorized person of the participating laboratory and/or the respective accreditation body.

In general, all information on participant performance shall not be disclosed to any third party unless prior agreement with the concerned participants has been obtained or applicable laws or regulations stipulate such disclosure. GLHK, the proficiency testing provider for this proficiency testing programme, shall also take into consideration local regulatory requirements for the disclosure of confidential information. GLHK may disclose any relevant information to Hong Kong Accreditation Service for accreditation purposes, with the consent/agreement obtained from participating laboratories through completion of the Registration Form / Sample Receipt Form / Result Proforma for this proficiency testing programme.

## 7. Contact

Participants may wish to contact the co-ordinator of the proficiency testing programme for any enquires (E-mail: [aplacpt.rice@govtlab.gov.hk](mailto:aplacpt.rice@govtlab.gov.hk)).

## 8. References

- [1] CODEX STAN 193-1995 “Codex General Standard for Contaminants and Toxins in Food and Feed”, 2016, CODEX Alimentarius.
- [2] COMMISSION REGULATION (EU) 2015/1006 “amending Regulation (EC) No 1881/2006 as regards maximum levels of inorganic arsenic in foodstuffs”, 2015, Official Journal of the European Union.
- [3] Reference to the dry mass correction procedures of NMIJ CRM 7503b.
- [4] ISO/IEC 17043:2010 “Conformity assessment – General requirements for proficiency testing”, 2010, Geneva, Switzerland.
- [5] ISO 13528:2015 “Statistical methods for use in proficiency testing by interlaboratory comparison”, 2015, Geneva, Switzerland.
- [6] W. Horwitz, *Anal. Chem.* 1982, **54**, 67A-76A “Evaluation of analytical methods used for regulations of food and drugs”.