Instructions for Participants

1. Objectives

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. At the APMP General Assembly and Related Meetings held in Taipei in November 2013, both APMP and APLAC agreed to establish the APMP-APLAC Joint PT Working Group as a formal infrastructure to provide more proficiency testing programmes with metrologically traceable reference values for performance evaluation purpose. To echo this new initiative, the Government Laboratory, Hong Kong (GLHK) proposed a new PT study for the organochlorine pesticides in ginseng root for 2016/17. The purpose of this study is to demonstrate the capability of participating laboratories in measuring organochlorine pesticides in a relatively complex food matrix/botanical material (e.g. ginseng root). Alpha-hexachlorocyclohexane (α-BHC, CAS No. 319-84-6) and gamma-hexachlorocyclohexane (Lindane, CAS No. 58-89-9), which are commonly used organochlorine pesticides for the growth of ginseng, are selected as the analytes in this PT study. The Commission Regulation of European Union sets up that the maximum residue level (MRL) required by for hexachlorocyclohexane (sum of isomers, except lindane) is 0.02 mg/kg and that for lindane is 1 mg/kg in ginseng [1]. The use of reliable methods for measurement of these organochlorine pesticides is important in safeguarding the quality of ginseng and related products and the public health.

The APMP-APLAC Joint Proficiency Testing Programme (APLAC T106) is coordinated by Government Laboratory of Hong Kong (GLHK). The objective of this study is to enable participating laboratories to demonstrate their competence on the measurement of the mass fractions of the two analytes (α-BHC and Lindane) in the proficiency test sample of ginseng root by various analytical techniques. The mass fractions of the analytes on a dry mass basis will be used for comparability purposes.

2. Analysis of the proficiency test sample

Participating laboratories will be provided with **ONE** sample bottle containing about **25 g** of ginseng root 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:

ginseng2016@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 at about -20 ^{o}C prior to analysis. The temperature of the bottle shall reach the room temperature (20 ± 5 ^{o}C) before opening.

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. The proficiency test sample should be mixed thoroughly before conducting the tests and the analysis shall be conducted with a recommended sample size of at least 1 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. The two measurands and the range of values to be expected for the proficiency test sample are given as follows:

Measurand	**Molecular weight**	**Mass fraction (expected range of values) (μg/kg)**
α-BHC	290.831	10 – 1000
Lindane	290.831	10 – 1000

It is noteworthy that wetting of test sample prior to extraction was crucial for complete extraction of the incurred analytes in dried food/plant matrices as learned from the key comparison CCQM-K95 [2]. For the sample pre-treatment, it is recommended that participants should wet each portion of the sample with an appropriate amount of water for sufficient time prior to extraction and subsequent measurement.

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 placed over anhydrous calcium sulphate (DRIERITE®) in a desiccator at room temperature for a minimum of 20 days until a constant mass is reached. 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 μg/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; and
- Participants should provide information about 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 Proforma electronically to GLHK (E-mail: ginseng2016@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.

4. Performance evaluation

The performance of the participating laboratories will be assessed using *z*-score, which is calculated as follows [3, 4]:

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

where x_i : the participant's result
x_{pt} : the assigned value*
σ_{pt} : the standard deviation for proficiency assessment estimated from the Horwitz equation [4, 5]

* Note: The Supplementary Comparison Reference Values (SCRVs) obtained from APMP supplementary comparison APMP.QM-S11 would be used as the assigned values for evaluating the performance of participants. This is in accordance with the ISO/IEC 17043 recommendations on the determination of assigned values for proficiency testing schemes [3].

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 (ζ), which is calculated as follows [4]:

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

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}

ζ-scores are interpreted as in the same way as z-scores using the same critical values of 2.0 and 3.0. ζ-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 [4]. 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	Event
November 2016	Call for Participation
9 December 2016	Deadline for registration
December 2016	Distribution of samples
31 March 2017	Deadline for submission of results
June 2017	Issue of interim report
December 2017	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, APLAC 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 laboratories. The code number assigned to a participant in the proficiency testing programme is only made known to the contact person/authorised 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: ginseng2016@govtlab.gov.hk).

8. References

[1] Commission Regulation (EC) No. 396/2005.
[2] CCQM-K95 Final Report, *Metrologia*, 2015, **52**, Tech. Suppl., 08007.
[3] ISO/IEC 17043:2010 "Conformity assessment – General requirements for proficiency testing", 2010, Geneva, Switzerland.
[4] ISO 13528:2015 "Statistical methods for use in proficiency testing by interlaboratory comparison", 2015, Geneva, Switzerland.
[5] W. Horwitz, *Anal. Chem.* 1982, **54**, 67A-76A "Evaluation of analytical methods used for regulations of food and drugs".