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 Proficiency Testing Working Group (PTWG) 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 on the determination of elements in food supplement for 2016/17. The purpose of this study is to demonstrate the capability of participating laboratories in measuring essential elements in a relatively complex food matrix/biological material (e.g. food supplements). Zinc, manganese, calcium and magnesium, which are the micronutrients essential for good health and are the elements commonly formulated in food supplements, are selected as the analytes in this PT study.

CODEX Alimentarius establishes the guidelines that apply to vitamin and mineral food supplements intended for use in supplementing the daily diet with vitamins and/or minerals [1]. Mineral food supplements are commercially available and marketed in forms of tablets, capsules, powders, solutions, etc. In cases where the intake from the diet is insufficient or where consumers consider their diet requires supplementation, mineral food supplements serve to supplement the daily diet. The amounts of the minerals should be properly declared in the labelling of the products. The use of reliable methods for measurement of minerals is important in safeguarding the quality of these products and the public health.

The APMP-APLAC Joint Proficiency Testing Programme (APLAC T107) is coordinated by GLHK. The aim of this study is to demonstrate the capability of participating laboratories in measuring the mass fractions of the elements (e.g. zinc, manganese, calcium and magnesium) at mg/kg levels in the proficiency test sample of food matrix/biological material (e.g. food supplement) by various analytical techniques.

2. Analysis of the proficiency test sample

Participating laboratories will be provided with **ONE** bottle containing about **25 g** of food supplement 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: aplacpt.supplement@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 four 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)
Zinc	1000 – 20000 mg/kg
Manganese	1000 – 10000 mg/kg
Calcium	10000 – 200000 mg/kg
Magnesium	10000 – 100000 mg/kg

Participants should also carry out the dry mass correction. For the determination of dry mass correction, a minimum of three separate portions (with a recommended sample size of about 1 g for each portion) 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 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 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.

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.supplement@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 [2, 3]:

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

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 [3, 4]

* Note: The Supplementary Comparison Reference Values (SCRVs) obtained from APMP supplementary comparison APMP.QM-S10 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 [2].

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 [3]:

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

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 [3]. 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**
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/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.supplement@govtlab.gov.hk).

8. References

[1] CAC/GL 55-2005 "Guidelines for vitamin and mineral food supplements", 2005, CODEX Alimentarius.
[2] ISO/IEC 17043:2010 "Conformity assessment – General requirements for proficiency testing", 2010, Geneva, Switzerland.
[3] ISO 13528:2015 "Statistical methods for use in proficiency testing by interlaboratory comparison", 2015, Geneva, Switzerland.
[4] W. Horwitz, *Anal. Chem.* 1982, **54**, 67A-76A "Evaluation of analytical methods used for regulations of food and drugs".