



APLAC Proficiency Testing Programme (APLAC T081)
Pesticides in Tea



Introduction

A CCQM key comparison CCQM-K95 “Mid-polarity Analytes in Food Matrix: Mid-Polarity Pesticides in Tea ” will be organized by the Consultative Committee for Amount of Substance (CCQM). A proficiency test programme “Pesticides in Tea” using the same material will be organized by the Asia-Pacific Metrology Programme (APMP) as a joint initiative of its Technical Committee for Amount of Substance (TCQM) and the Developing Economics’ Committee (DEC). The initiative was first discussed in 2009 and this comparison for pesticides in green tea leaves was proposed and endorsed at the APMP TCQM and DEC meetings in Pattaya, Thailand in 2010. The CCQM key comparison and its associated pilot study (CCQM-K95 and CCQM-P136) and the APMP DEC proficiency test (APMP PT 11-02) will be conducted in parallel.

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, APMP will also collaborate with the Asia Pacific Laboratory Accreditation Cooperation (APLAC). The same study material will be used in an APLAC proficiency test that will concurrently be conducted in parallel with the CCQM –K95 and APMP PT 11-02.

The proposal for organizing this APLAC PT programme was first proposed in Bali, Indonesia in 2009 and was further discussed at the APLAC PT Committee meeting at Osaka, Japan in December 2010. At this meeting, it was agreed that this programme would be organized in 2011. Subsequently, a formal proposal for organizing this PT program was circulated to the APLAC PT Committee members and approval was granted. An APLAC PT program code, APLAC T081, was assigned to this program.

Organisers of the APLAC PT Programme

Government Laboratory (GLHK) is the proficiency testing provider and Hong Kong Accreditation Service (HKAS) is the proposer.



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Responsibilities

GLHK is responsible for preparing, packaging and dispatching samples, performing homogeneity and stability tests, collecting test results from participating laboratories, conducting statistical analysis of data, handling participants' queries and issuing interim and final reports.

HKAS is responsible for proposing this program for approval by the APLAC Proficiency Testing Committee, inviting participants, circulating the draft report and final report to participants and acting as a contact point between APLAC, participating accreditation bodies / participants and GLHK.

Application Fee

Free of charge.

Selection of Participants

APLAC members as well as other non-APLAC members (EA and IAAC members as well as non-affiliated accreditation bodies) will be invited to participate in the programme. Invitations will be circulated to all APLAC members and other non-affiliated ILAC accreditation bodies, as well as the PT program coordinators of the respective regional accreditation cooperation 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 preferably limited to 100. Therefore, a restriction on the number of participating laboratories from each accreditation body may need to be imposed. When the number of enrolments exceeds the limit, participants will be selected on a first come first served basis and the organisers will, as far as possible, allow at least one laboratory to participate in this programme from each accreditation body.



Description of Materials

About 10 kg of dried green tea leaves purchased from the local market were confirmed to contain incurred pesticide residues. The material was powdered, sieved, homogenized and disinfected by γ -irradiation at a dose of about 1 kGy. The homogenized powder, about 20 g portion each, was independently dispensed into clean amber bottles. The approximate ranges of residual pesticides are as follows:

Pesticide	Approximate Quantity ($\mu\text{g}/\text{kg}$)
β -Endosulfan	100 to 1000
Endosulfan Sulphate	100 to 1000

Instructions for participants

The sample will be delivered with cold pack. Each participant will receive one bottle (about 20 g) of the sample. It is recommended that the material should be stored below 20 °C and preferably at ~ 4°C to avoid analyte degradation.

For the determination of dry mass correction, a minimum of three separate portions (recommended size to be 1 g each) of the sample should be taken and placed over anhydrous calcium sulphate in a desiccator at room temperature for a minimum of 10 days until a constant mass is reached. Dry mass correction should be carried out at the same time as the test sample portions are analysed. Results on dry mass basis should be reported

Homogeneity & Stability Tests

Not less than ten samples were taken randomly from the prepared bottles of samples and analyzed in duplicate for determining the sample inhomogeneity. Random samples will be analyzed in duplicate at room temperature (about 20 °C) and at elevated temperatures (about 30, 35 and 40 °C respectively) for monitoring the stability of analytes throughout the course of study.



Reporting Results

Participants will be provided with ONE sample bottle each containing about 20 g of testing material. Participants are asked to determine and report the concentrations (in $\mu\text{g}/\text{kg}$) of the above two organochlorine pesticides on a dry mass basis in the given testing material with their preferred methods. Test results, expanded measurement uncertainties and other technical details should also be reported in the given result sheets to the organizers. If the determination has been carried out in duplicate or triplicate, laboratories should report the mean result for performance assessment.

Participants should be aware that any submitted result is considered final and accordingly such data and units should be thoroughly checked before submission.

Performance Assessment

Performance of the participating laboratories is assessed using z-score which is calculated as:

$$z = \frac{x_i - x}{\sigma}$$

where x_i = reported result of individual participant
 x = assigned value*
 σ = standard deviation for proficiency assessment estimated from the Horwitz Equation

Note: * The assigned values will be provided by the reference values obtained from the corresponding CCQM Key Comparison (CCQM K-95). This is in line with the ISO/IEC 17043 recommendations on the determination of assigned values for PT scheme.

z-Score is commonly interpreted as:

- | | | |
|-------|---------------|----------------|
| (i) | $ z \leq 2$ | Satisfactory |
| (ii) | $2 < z < 3$ | Questionable |
| (iii) | $ z \geq 3$ | Unsatisfactory |



Laboratories having a $|z|$ score equal to or larger than 3 shall thoroughly investigate their results for the discrepancy and those having a z-score in the range $2 < |z| < 3$ are also encouraged to review their results.

Submission of Results

Participants should submit their results electronically to GLHK on or before the deadline. Results submitted after the deadline will not be accepted. Under no circumstances, correction or adjustment of analytical data will be accepted after the issuance of the interim report.

Issuance of Reports

Upon completion of data analysis, an interim report will be issued to participants and/or their respective accreditation bodies for checking the correctness of analytical data. The draft final report will then be prepared and submitted to APLAC PT Committee for comment and approval. Upon approval by the APLAC PT Committee, an electronic copy of the Final Report will be distributed to participants and their respective accreditation bodies.

Final report, part of the final report or its summary may be posted onto the website of GLHK for public interests.

Programme Schedule

Event	Period
Preparation of sample	Jan – Mar 2011
Homogeneity testing	Mar 2011
Stability testing	From Mar 2011
Invitation of participants	July /Aug 2011
Deadline for registration	30 Sept 2011



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Dispatch of samples	Nov 2011
Submission of results	28 Feb 2012
Statistical analysis of results	Mar 2012
Distribution of interim report	Mar 2012
Issuance of final report	Aug 2012

Confidentiality

The organizers (HKAS and GLHK) strive to maintain strict confidentiality with respect to composition of the test sample distributed and the performance of all participating laboratories. To preserve this confidentiality, participants receive reports giving all results for that assessment but without identifying individual laboratories. The code number assigned to a participant in this programme is only made known to the contact person or authorized person of his laboratory, the organizers (GLHK, HKAS and APLAC) and/or the respective accreditation body/regional accreditation body cooperations.

This 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 this program.

Contacts

If you have any query or comment on the proposal, please contact Mr. W. W. Wong of HKAS at wwwong@itc.gov.hk.