Asia Pacific Laboratory Accreditation Cooperation
Proficiency testing scheme (APLAC T084)
Organochlorine pesticide residues in chicken fat

Jointly coordinated by:
Bureau of Quality and Safety of Food (BQSF) & Bureau of Laboratory Quality Standard (BLQS)
Department of Medical Sciences, THAILAND

Protocol

1. Introduction

Proficiency testing (PT) is an evaluation of participant performance against pre-established criteria by means of interlaboratory comparison [1]. The organochlorine pesticides are well-known persistent pesticides, which have been banned in many countries. Due to their properties of lipophilicity and bio-accumulation in food chain, they are targeted analytes which are routinely tested for food safety.
The aims of the study were to:

- assess the accuracy in the measurement of organochlorine pesticides in chicken fat;
- develop participants' practical application of traceability and measurement uncertainty and provide information that will assist their uncertainty estimates;
- provide accreditation bodies (AB) with objective evidence of laboratory performance.

2. Organizers

The Bureau of Quality and Safety of Food (BQSF) is the PT provider. BQSF takes responsibility for all tasks in the development and operation of the PT scheme, including preparation and distribution of PT items, data analysis and evaluation of results. Mrs Kanokporn Atisook has been assigned as the coordinator of the PT scheme.

The Bureau of Laboratory Quality Standard (BLQS) is the proposer. BLQS is responsible for proposing the PT scheme for approval by APLAC PT committee, inviting participants, circulating the interim report and the final report to participants and acting as a contact point between APLAC, accreditation bodies/participating laboratories and BQSF.

Address of organizers:
1. Bureau of Quality and Safety of Food (BQSF)
Department of Medical Sciences (DMSc)
Ministry of Public Health
88/7 Tiwanon Rd.
Nonthaburi 11000 THAILAND
2. Bureau of Laboratory Quality Standard (BLQS)
Department of Medical Sciences (DMSc)
Ministry of Public Health
88/7 Tiwanon Rd.
Nonthaburi 11000 THAILAND

3. Scheme coordinator

PT provider: The provider is responsible for all aspects of the testing schemes.

Mrs. Kanokporn Atisook
Medical Scientist, Expert level
Bureau of Quality and Safety of Food (BQSF)
Department of Medical Sciences (DMSc)
Ministry of Public Health
88/7 Tiwanon Rd.
Nonthaburi 11000 THAILAND
Tel/Fax +662 951 1021 Email: kanokporn.a@dmsc.mail.go.th

APLAC T084 coordinator: a contact point between APLAC, accreditation bodies/participating laboratories and BQSF.

Mrs. Chomchailai Sinthusarn
Medical Scientist, Expert level
Bureau of Laboratory Quality Standard (BLQS)
Department of Medical Sciences (DMSc)
Ministry of Public Health
88/7 Tiwanon Rd.
Nonthaburi 11000 THAILAND
Te l +662 951 1455 /Fax +662 965 9755 Email: chomchailai.s@dmsc.mail.go.th

4. Fee of participation

Free of charge

5. Selection of participants

APLAC members as well as other non-APLAC accreditation bodies will be invited to participate in the scheme. Invitation will be sent to all APLAC members and other accreditation bodies. Accreditation bodies will be asked to nominate laboratories for participation and indicate the accreditation status of the nominated laboratories. The number of participating laboratories shall be limited to 50. The organizers will allow maximum 4 laboratories from each accreditation body to participate in this PT scheme.

6. Test items

Participating laboratories will be provided with one vial containing about 6 g of a homogenate chicken fat spiked with selected pesticides.

Preparation

Two kilograms of liquefied chicken fat (previously analyzed and showed “not detected” result) are bulked and mixed together. One kilogram is used for “Blank” and the other is used for “Spiked”. For the first portion “Blank”, aliquots of about 6 g are packed into amber vial and labeled as “BF” for “Blank chicken fat” and then stored in refrigerator. For the second portion “Spiked”, known amount of organochlorine pesticide standards are added and mixed, aliquots of about 6 g are packed into vial and labeled as “SF” for “Spiked chicken fat” and then stored in refrigerator.

Homogeneity testing

The test item is tested for homogeneity by laboratory of Bureau of Quality and Safety of Food (BQSF), DMSc. Not less than 10 vials will be randomly selected and analyzed in duplicate for determining the sample inhomogeneity for each analyte. Evaluation of results is keeping with those recommended in the International Harmonized Protocol [2].

Stability testing

- Before distribution of test items, not less than 3 vials will be randomly selected and stored in elevated temperature about 45 ± 5°C for at least 5 days. Then the conditioned test items will be analyzed in duplicate for monitoring sample instability.
- On the last day of deadline for returning results, not less than 3 vials will be randomly selected from the refrigerator and analyzed in duplicate for monitoring sample instability.
- Assessment of adequacy of stability was calculated by comparing average of detected results obtained from homogeneity testing with the average of those obtained from stability testing. (ISO 13528: 2005) [3].

Distribution of test items and documents

One vial containing about 6 g of spiked chicken fat together with;

- One vial containing about 6 g of blank chicken fat for negative control and recovery study
- Instructions to Accreditation Bodies (ABs)
- Participating Laboratories Nomination Form
- Receipt Form for Accreditation Bodies (ABs)
- Instructions to participating Laboratories
- Receipt Form for participating Laboratories
- Results sheet

They are sent to participating ABs. Test items are packaged to minimize deterioration in transit. Participating ABs must provide BLQS with any import or quarantine permits that might be necessary.

7. Methods of analysis

Participants are instructed to perform the analysis using their normal test methods and report a single result, together with an associated uncertainty, for each pesticide that is detected. The reported results should not be corrected for recovery, however participants are asked to report the percent recovery if it has been determined. A list of organochlorine pesticides which possible spiked into the samples are aldrin, cis-chlordane, trans-chlordane, dieldrin, α-endosulfan, β-endosulfan, endosulfan sulfate, endrin, HCB, α-HCH, γ-HCH, heptachlor, heptachlor epoxide, oxychlordane, p,p'-DDE, p,p'-DDD and p,p'-DDT. Participants may choose to test for all or for only some of these and may report which compounds are not tested in their scope of analysis.

8. Reporting and submission of results

Participants should complete the "Result Report Sheet". The manner of reporting test results are as follows:

- Report amount of analytes found in μg/kg, as received (i.e. on a whole basis), uncorrected for recovery
- For each analyte, the single result together with an associated uncertainty should be reported
- Participants should provide information about methods of analysis, percent recovery and limit of quantitation (LOQ).

Participants should be aware that any submitted results are considered final and accordingly such results and units should be thoroughly checked before submission. Results submitted after deadline will not be accepted. Under no circumstances, correction or adjustment of analytical data will be accepted after the issue of the interim report.

9. Establishing the assigned value and target standard deviation

The assigned value is the value which participants' results are compared, and must be the best available estimate of the true concentration of analyte.

For this PT scheme, the assigned values, X are established by higher order measurement (e.g. isotope dilution mass spectrometry, IDMS) and by measurement alongside a reference material traceable to an international standard which are known as reference values.

The value of target standard deviation (σ) determines the limits of satisfactory performance which derives from the appropriate form of the Horwitz equation [4]. This equation predicts a standard deviation from a given concentration, c, and requires c to be expressed as a dimensionless mass ratio. It follows therefore that to express the dimensionless standard deviation predicted by the equation in the original concentration units it must be divided by the relevant mass ratio:

i) for analyte concentrations < 120 μg/kg

$$\sigma = \frac{0.02\,c}{mr}$$

ii) for analyte concentrations ≥ 120 μg/kg and ≤ 13.8%

$$\sigma = \frac{0.02\,c^{0.8495}}{mr}$$

iii) for analyte concentrations > 13.8%

$$\sigma = \frac{0.01\,c^{0.5}}{mr}$$

where c = concentration, i.e. the assigned value, X expressed as a dimensionless mass ratio
e.g. ppb or μg/kg is 10^{-9} or % is 10^{-2}
mr = dimensionless mass ratio, e.g. ppb or μg/kg is 10^{-9} or % is 10^{-2}

10. Performance evaluation

Participants are requested to report their results with the associated measurement uncertainty and additional information on analysis method used.

The participants' results are evaluated using the *z* score as follow:

$z = (x - X)/\sigma_p$

where σ_p is target standard deviation

x is the participant's result

X is the assigned value

Evaluation of performance

$|z| \leq 2.0$ indicates "satisfactory" performance

$2.0 < |z| < 3.0$ indicates "questionable" performance

$|z| \geq 3.0$ indicates "unsatisfactory" performance

11. Issue of reports

An interim report will be issued to participants and their respective accreditation bodies for checking the correctness of results submitted. The draft final report will then be prepared and submitted to APLAC PT Committee for comments and approval. Upon approval, an electronic copy of the final report will be distributed to the accreditation bodies to inform the participants they nominated.

12. Proposed program schedule

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

Proposed time schedule	Phase
October 2012	Call for participation
November 2012	Deadline for registration
December 2012	Distribution of test items
February 2013	Deadline for submission of results
March 2013	Interim report for comments
May-June 2013	Draft final report for comments
End of July 2013	Issue of the final report

13. Confidentiality and Ethical considerations

The concerned parties (APLAC, BQSF and BLQS) strive to maintain strict confidentiality with respect to composition of the PT test item distributed and performance of all participants. To preserve the confidentiality, participants will receive reports giving all results for assessment but without identifying individual laboratories. The identity of participants is protected by means of a laboratory code. The code number assigned to a participant in the proficiency testing scheme is only made known to the contact person of the participating laboratory and/or the respectively accreditation body.
The PT scheme is conducted in the belief that participants perform the analysis and report results with scientific rigor. However PT organizer will take steps to prevent collusion or falsification of results by participants. Where any collusion or falsification is proven, the results of the participant for the PT concerned will be eliminated and the laboratory manager will be notified.

14. References

1) ISO/IEC 17043: 2010. Conformity assessment – General requirements for proficiency testing.

2) Thompson M., Ellison S.L.R., Wood R., 2006. The International Harmonized Protocol for the proficiency testing of analytical chemistry laboratories (IUPAC Technical Report), in Pure and Applied Chemistry, Vol.78, No.1, pp. 145-196.

3) ISO 13528: 2005. Statistical methods for use in proficiency testing by interlaboratory comparison

4) Thompson M., 2000. Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing. Analyst 125, 385-386.