

Protocol for Proficiency Testing Scheme of Metals in Food BQSF ME 04-64 (Fish)

1. Introduction

Proficiency testing (PT) is an evaluation of the performance of laboratories for specific tests or measurements and monitoring laboratories' continuing performance, by comparing analytical results between participating laboratories. Regular participation of the PT scheme is as a tool to reveal additional confidence in laboratory performance not only for laboratories but also their customers.

The ASEAN Food Reference Laboratory (AFRL) for Heavy Metals and Trace Elements is part of the Bureau of Quality and Safety of Food (BQSF), Department of Medical Sciences (DMSc), Ministry of Public Health, Thailand. In order to assist in the development of ASEAN member countries' capacities, the AFRL for Heavy Metals and Trace Elements has organised the PT schemes, financed by Thai Government budget, for testing laboratories in AMSs since 2005 until present.

2. Terminology and abbreviation

- Proficiency testing scheme: proficiency testing designed and operated in one or more rounds for a specified area of testing, measurement, calibration or inspection.
- Proficiency testing item (or PT sample): sample, product, artefact, reference material, piece of equipment, measurement standard, data set or other information used for proficiency testing.
- Proficiency testing provider: organisation which takes responsibility for all tasks in the development and operation of a proficiency testing scheme.
- Scheme coordinator: one or more individual for which a proficiency testing scheme is provided through a contractual arrangement.
- Assigned value: value attributed to a particular property of a proficiency test item.

3. PT Provider

AFRL for Heavy Metals and Trace Elements
Bureau of Quality and Safety of Food (BQSF)
Department of Medical Sciences (DMSc), Ministry of Public Health
Tiwanon Road, Nonthaburi 11000 Thailand

4. Scheme coordinators

Mr. Sirichai Sunya	Medical Scientist, Professional level
Miss Panawan Kluengklangdon	Medical Scientist, Senior professional level

5. Purpose of the scheme

The aim of the scheme is to evaluate laboratory performance for analysis of metals in food. A number of participating laboratories are limited on a first-come, first-served basis up to 60 participants (If there are registered participants fewer than 12 laboratories, the PT Scheme will not be organised for that year).

6. Participation fee

Free of charge

7. Timeable for PT scheme

The time schedule for the various phases of the PT scheme is proposed as follows:

Time schedule	Phase
29 January 2021	Deadline for submission of the registration form
16 February 2021	Distribution of the test item
30 April 2021	Deadline for submission of PT results
30 June 2021	Issue of the final report

8. PT samples

Food sample (fish) is fortified with total As, Cd, Cr, Cu, Fe, Pb, Mn, total Hg, Ni and Zn of which the concentration range will be provided in the PT's instruction

9. Handling of PT samples

9.1 Preparation of PT samples

Fish, Nile tilapia (*Oreochromis niloticus*), is filleted and milled; and the concentrations of metals in the starting fish samples are preliminary quantified. The spiking solutions are prepared starting from the certified stock solutions. The bulk material is prepared by adding deionised water and maintained under magnetic stirring before, during and after the addition of the spiking solution. The spiked fish samples are then freeze-dried, milled using an appropriate blender to sufficient finesse and passed through a 500-mesh sieve. The spiked fish samples are homogenized, weighed and stored in a seal aluminium foil bag at ambient temperature. Participating laboratories will be provided with one sample contained in a seal aluminium foil bag in which contains approximately 15 g of the fish sample.

9.2 Homogeneity test

The test items are tested for sufficient homogeneity by the laboratory of Bureau of Quality and Safety of Food (BQSF), DMSc which has been accredited in the field of food testing in compliance with ISO/IEC 17025: 2017. Ten bottles are randomly selected and analysed in duplicate for testing homogeneity of each analyte. Assessment of the obtained results is conducted, using appropriate statistical approaches based on ISO 13528:2015; Annex B.

9.3 Stability test

Assessment of adequacy of stability is performed at (or one-day before) the date of sample dispatch and at the date of (or after) the deadline of result submission. Three bottles of test items are randomly selected at the day of sample dispatch and placed in the parcel at ambient temperature until the deadline of result submission. Then these three bottles of test items are analysed in duplicate by the

same testing laboratory and by the same methods as 9.2 Homogeneity test. Assessment of the obtained results is conducted, using appropriate statistical approaches based on ISO 13528:2015; Annex B.

9.4 Assigned value

Three ways of determining the assigned value (x_{pt}) can be carried out with the following approaches:

- Consensus value from participant results is estimated using a robust analysis (algorithm A) based on ISO 13528:2015; Annex C;
- Reference value from a single laboratory is determined using a series of tests, in one laboratory, on proficiency test items and the certified reference material (CRM), using the same measurement method, and under repeatability conditions, based on ISO 13528: 2015;
- Consensus value from expert laboratories is determined using an interlaboratory comparison study with expert laboratories according to ISO 13528: 2015; 7.6.

10. Method of analysis

Participants are instructed to perform the analysis using their routine test methods (standard method recommended).

11. Evaluation of participants' results

An assigned value is the value to which participants' results are compared, and must be the best available estimate of the true concentration of analyte(s). The value of standard deviation of proficiency assessment (σ_{pt}) determines the limits of satisfactory performance which can derive from a general model (e.g. Horwitz equation) or from robust standard deviation or from approaches with appropriate statistics based on ISO 13528:2015. The approach used for evaluating the individual laboratory performance is the z- score calculated by the following equation:

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

Where

x_i denotes the reported result

x_{pt} denotes the assigned value for the proficiency test item;

σ_{pt} denotes the standard deviation for proficiency assessment (i.e. target standard deviation)

Another approach for evaluating the individual laboratory performance, z' -score, can be used when there is concern about the uncertainty of an assigned value $u(x_{pt})$. The uncertainty can be taken into account by expanding the denominator of the performance score according to ISO 13528:2015. The conventional interpretation of z-scores/ z' -scores is as follows:

- $|z|$ or $|z'| \leq 2.0$ indicates "satisfactory" performance and generates no signal
- $2.0 < |z|$ or $|z'| < 3.0$ indicates "questionable" performance and generates a warning signal
- $|z|$ or $|z'| \geq 3.0$ indicates "unsatisfactory" performance and generates an action signal

12. PT report

An electronic copy of the final report will be distributed to the participants. This report shows only laboratory codes, z-scores/z'-scores and assigned values in the form of table or graph as appropriate.

13. Confidentiality and ethical consideration

- 13.1 Assigned value will not be revealed to members until the final PT report is sent to the participants. In addition, each member will receive a unique laboratory code and no laboratory code will be specified along with the laboratory name in order to protect the confidentiality of the members. When the summary of the results or a part of results are used for public communication, the name of participating laboratories shall not be mentioned in any case.
- 13.2 The participating laboratories should perform the analysis and report results with scientific rigor and honesty, where the analytical results should be neither communicated to others nor asked from other participants. Therefore, the members can take full advantage of the PT results to meet its objectives with greatest benefits.

14. Communication with participants

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