



กรมวิทยาศาสตร์การแพทย์  
Department of Medical Sciences

PT Protocol ME  
Protocol for Proficiency  
Testing Scheme of Metals  
in Food  
Revision No. 01



หน้า 1/7

วันที่อนุมัติใช้ 2-6 มิ.ย. 2563

ผู้แก้ไข

(นายศิริชัย สัตถุญะ)

ผู้ทบทวน

(นางสาวจิตผกา สันทัดรบ)

ผู้อนุมัติใช้

(นายอรัญ ทนันทิติ)

## Protocol for Proficiency Testing Scheme of Metals in Food

### 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), the 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, which the scheme coordinator sends to participating laboratories, must have a suitable homogeneity and stability.**
- 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 coordinator and team

Mr. Sirichai Sunya	<b>Medical Scientist, Professional level (Scheme coordinator)</b>
Ms. Panawan Kluengklangdon	<b>Medical Scientist, Senior professional level</b>

## 5. Purpose of the scheme

The aim of the scheme is to evaluate laboratory performance for analysis of metals (e.g. cadmium, lead, total mercury, total arsenic, inorganic arsenic, copper, zinc, iron, manganese, selenium) 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 less 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 yearly proposed as follows:

Time schedule	Phase
August <b>to</b> December	Call for participation
January to mid-February	Deadline for registration
mid-February to mid-March	Distribution <b>of test items</b>
April <b>to</b> May	Deadline for submission of <b>PT</b> results
June <b>to</b> July	Issue of the final report

(Exact dates of each activity will be shown in the Registration Form)

## 8. PT samples

### 8.1 Test Solution

Test solution is a mixture of standard solutions of metals (e.g. cadmium, lead, total mercury, total arsenic, inorganic arsenic, copper, zinc, iron, manganese, selenium). **In addition, ranges of each analyte will be provided in the PT's instruction.**

### 8.2 Food sample

Food samples (e.g. milk powder, dry seaweed, rice, fish, tea) are naturally contaminated by metals (e.g. cadmium, lead, total mercury, total arsenic, inorganic arsenic, copper, zinc, iron, manganese, selenium). In case of absence or low level of desired metals, the fortification of the desired metals in food samples will be considered. **In addition, ranges of each analyte will be provided in the PT's instruction.**

## 9. Handling of PT samples

### 9.1 Preparation of PT samples

Test solution is prepared by adding standard solutions of metals with pre-defined concentrations to deionized water, maintained under magnetic stirring before, during and after the addition of the spiking

standard solutions. Nitric acid is added in the mixture to reach a pH < 2. **One to two** mL of the mixture is filled in a 2 mL HDPE tube with **a label indicating a code number, then** stored in the refrigerator between 2 and 10° C until sending to the participants.

Food sample are milled; and the concentrations of metals in the starting food samples are quantified. In case of fortification with metals, the spiking solutions are gravimetrically 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 solution of food samples is then freeze-dried, milled using an appropriate blender to sufficient finesse and passed through a 200-mesh or a 500-mesh sieve. The spiked food samples are homogenized, weighed and stored in airtight plastic containers at room temperature or in the refrigerator between 2 and 10° C (depending on the food matrices) until needed. Participating laboratories will be provided with one sample contained in a **high-density** polypropylene bottle (**HDPE**). Each bottle contains approximately 10-30 g of the food 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. The analytical methods are as follows:

- Arsenic (total) (As): AOAC (2019) 986.15 & 999.10;
- Inorganic Arsenic (iAs): In-house method SOP 20 02 325 based on J. Food hyg. Soc.Jpn. 2007, Vol 49, No.2;
- Cadmium (Cd) and Lead (Pb): AOAC (2019) 999.10;
- Mercury (Hg): In-house method SOP No.20 02 030 based on AOAC (2019) 997.15 หรือ In-house method SOP No. 20 02 008 based on J. Analyst, Vol.108, 1983, p.1313-1317;
- Lead (Pb), Cadmium (Cd), Arsenic (As), Copper (Cu), Iron (Fe), Zinc (Zn), Tin (Sn), Nickel (Ni), Manganese (Mn), Chromium (Cr): In-house method SOP 20 02 365 based on AOAC (2019) 2000.08 by ICP-OES Technique.

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 room temperature for the period of delivery time, and then kept in the refrigerator between 2 and 10° C 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) and report a single result of each analyte in three significant figures (concentrations in mg/L for the test solution and/or in  $\mu\text{g}/\text{kg}$  dry mass for the food sample). In addition, moisture content in food sample shall be determined for converting results to dry mass basis. The recommended drying method will be provided in the PT's instruction.

#### 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 ( $\sigma_{pt}$ ) 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;
- $\sigma_{pt}$  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. This approach can be applied when:

- For a homogeneity check, the between-sample standard deviation ( $s_s$ ) compares with the standard deviation for proficiency assessment ( $\sigma_{pt}$ ) and  $s_s > 0.3 \sigma_{pt}$ ;
- For a stability check, the difference of the averages of measurements obtained from the stability test and the homogeneity test is higher than  $0.3 \sigma_{pt}$ ;
- The uncertainty value obtained when determining the assigned value is higher than  $0.3 \sigma_{pt}$ .

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

Where

$u$  denotes the between-sample standard deviation ( $s_s$ ) and/or the values of standard uncertainty from the stability test or the assigned value

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, a 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

Mr. Sirichai Sunya (Scheme coordinator)  
Bureau of Quality and Safety of Food,  
**Department of Medical Sciences, Ministry of Public Health**  
**Tiwanon Road, Muang district, Nonthaburi 11000 Thailand**  
Tel: +662 951 0000 ext 99895  
Email: [sirichai.s@dmsc.mail.go.th](mailto:sirichai.s@dmsc.mail.go.th) or [bqsf\\_pt@dmsc.mail.go.th](mailto:bqsf_pt@dmsc.mail.go.th)