กรมวิทยาศาสตร์การแพทย์
DEPARTMENT OF MEDICAL SCIENCES

PROFICIENCY TESTING PROGRAM 2017

Scheme Code D6001R1

Assay by High Performance Liquid Chromatography

Protocol No. D6001R1

Organized by

Bureau of Drug and Narcotic
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1. ORGANIZER

Scheme Provider:

Bureau of Drug and Narcotic (BDN)
Department of Medical Sciences
88/7 Tiwanon Road, A. Muang, Nonthaburi 11000 THAILAND
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Scheme Coordinator:

Ms. Siriphorn Laomanacharoen
Pharmacist, Senior professional level
Email: pt_bdn@hotmail.com; pts.bdn@gmail.com
The scheme coordinator is responsible for all activities of the PT scheme.

2. OBJECTIVE

The objective of this Proficiency Testing (PT) scheme is to demonstrate that participants can accurately perform analysis by using High Performance Liquid Chromatography (HPLC) technique. The scheme is based on the analysis of pharmaceutical products, hydrochlorothiazide tablet, according to the method from the 39th revision of the United States Pharmacopeia (USP 39) on Hydrochlorothiazide Tablets monograph.

3. SUBCONTRACT

BDN has no policy to subcontract in any activity of this proficiency testing scheme.

4. SCHEDULE OF PT SCHEME

Schedule for the PT scheme activities is as follows:

Activity	Schedule
Call for participation	December 2016
Distribution of samples	March 2017
Deadline for submission of results	19 May 2017
Interim report for comments	July 2017
Final report	September 2017

5. REGISTRATION

Registration Fee:

Local participants	5,000.- Baht
Oversea participants	200.- USD (Including shipping handling)

Payment:
Local participants-
The payment should be made by Teller payment system at any branches of Krung Thai Bank Public Company Limited.

Oversea participants-
The payment should be made by bank transfer to

Account name:	Bureau of Drug and Narcotic
Account number:	142-1-12831-4
Bank name:	Krung Thai Bank Public Company Limited
Branch:	Ministry of Public Health, Nonthaburi
Swift code:	KRTHTHBK

Method and Deadline for Registration:

Participants should register online via the website **http://www.bdn.go.th/pt** within ***31 January 2017***. Registration by other means e.g. fax, Email will not be accepted.

Terms and Conditions:

1. BDN reserves the right to occasionally delay the issue of PT program or use an appropriate substitute test material with prior warning to participants if the planned PT sample is not available according to the schedule of PT scheme.
2. The registration fees are nonrefundable.

More information can be viewed on the website http://www.bdn.go.th/pt.

6. CRITERIA FOR PARTICIPATION

Participants should have competency to perform the analysis of hydrochlorothiazide tablets by High Performance Liquid Chromatography (HPLC) and use this technique in their routine work.

7. NUMBER AND TYPE OF PARTICIPANTS

Number of participants: 20 - 60

Type of participants: Quality control laboratories in pharmaceutical manufacturers and other testing laboratories

8. PROFICIENCY TESTING SAMPLE

The sample of this scheme is pharmaceutical product, hydrochlorothiazide 50 mg tablet.

9. HANDLING OF PT SAMPLE

Participants will receive (i) thirty tablets of hydrochlorothiazide 50 mg tablet, (ii) a bottle of reference standard containing about 120 mg of hydrochlorothiazide reference standard, (iii) a bottle containing about 100 mg of chlorothiazide for system suitability test, and (iv) a Testing protocol.

PT samples are packed in aluminium foil and distributed in ambient condition. The PT sample should be stored at room temperature (below 30°C), whereas reference standard and chlorothiazide should be stored in the refrigerator between 2-8°C. All of them should be protected from light and humidity until analysis.

PT samples are sent by express mail for local participants and by courier for oversea participants. It is the responsibility of the participants to contact BDN if they have not received the PT sample within the time schedule. In case of international transport for oversea participants, BDN cannot be responsible for any delays from the custom clearance. Upon receipt of PT sample, participants are requested to check physical conditions of PT sample as well as other substances provided, complete a PT sample acknowledgement form via the website http://www.bdn.go.th/pt.

10. TEST METHOD

Participants should determine the percentage content labeled amount of hydrochlorothiazide ($C_7H_8ClN_3O_4S_2$) and report the results with **one digit after the decimal separator**. Analysis is based on the assay method specified in USP 39 on Hydrochlorothiazide Tablets monograph using HPLC with the following chromatographic conditions.

HPLC Column: 4.6-mm × 25-cm; packing L1 (Octadecyl silane chemically bonded).

Detection : UV detector at 254 nm.

Details of the method are described in the testing protocol which will be enclosed with PT sample. PT sample as well as analytical procedure should be handled and operated in the same manner as performing routine work.

11. ASSIGNED VALUE

The assigned value used to calculate z score is based on the consensus value from participants using the robust mean calculated according to robust analysis:Algorithm A in Annex C of ISO 13528:2015-Statistical methods for use in proficiency testing by interlaboratory comparisons.

12. STANDARD DEVIATION FOR PROFICIENCY ASSESSMENT

Standard deviation for proficiency assessment used to calculate z scores is set as 2.0% that corresponds to the level of performance and the expected precision of the test method or techniques and according to fitness for purpose.

13. PERFORMANCE EVALUATION

Participants will be assessed on the differences between their results and the assigned value. The z score is used for the performance evaluation as

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

z_i = z score

x_i = measurement result from participant

x_{pt} = assigned value

σ_{pt} = standard deviation for proficiency assessment

The interpretation of z score is designated as follows.

$|z| \leq 2.0$: acceptable

$2.0 < |z| < 3.0$: warning signal

$|z| \geq 3.0$: unacceptable

For warning signal or unacceptable results, it is recommended that participants investigate root causes and take necessary corrective actions.

14. POTENTIAL MAJOR SOURCES OF ERRORS

1. Suitability of analytical balance used and weighing procedure
2. Accuracy of sample and reference standard solution preparation
3. Suitability of chromatographic system
4. Calculation
5. Moisture uptake due to inappropriate sample handling

15. REPORT

An interim report is issued to provide each participant with an early indication of performance. In general, the interim report is issued about two months after the deadline for submission of results via the website http://www.bdn.go.th/pt. Participants are requested to check and review for any correction and/or comment. The final report is issued via the website www.bdn.go.th/pt after the correction and/or comment of interim report has been completed.

The report includes the following information.

- Introduction: general description of PT scheme
- Name and contact details of proficiency testing provider and scheme coordinator
- Participation: information of participating laboratories
- PT sample: description, sample preparation
- Homogeneity and stability assessment
- Assigned value, including measurement uncertainty and standard deviation of proficiency assessment
- Results: result tables including statistic summary data, z scores and bar chart of z scores
- Discussion of results: conclusion of overall performance and comments on participants' performance
- Potential major sources of errors

16. CONFIDENTIALITY, COLLUSION AND FALSIFICATION OF RESULTS

The identity of participants is protected by means of laboratory code which is randomly assigned in each PT round. These codes are confidential and are not disclosed to other persons unless agreed by the participant for a regulatory or recognition purpose. Participants can access their personal information in the website http://www.bdn.go.th/pt by using username and password which can keep their information confidentially. For security purpose, participants are recommended to change username and password in case of changing the responsible person who can access the information in the website.

This PT scheme is conducted in the belief that participants will perform the analysis and report results with scientific professional. Where any collusion between participants

or falsification of results is proven by BDN, the result of that participant for the PT round concerned will be cancelled for performance evaluation.

17. LOST OR DAMAGED OF PROFICIENCY TESTING SAMPLE

In case of lost or damaged PT sample, participants should immediately inform BDN. The damaged PT sample should be returned to BDN. Replacement will be arranged if the PT samples are proved to be lost or considered not suitable for analysis. The deadline of submitting result will be extended to appropriate date if necessary.