Statistical Evaluation Throughout a PT programme: ISO 13528:2015

Mahidol University

Kunchit Judprasong

Institute of Nutrition, Mahidol University, Salaya,
Putthamonthon 4, Nakorn Pathom 73170
Tel. : 02 800 2380 Ext 324   Fax. : 02-441-9344
Kunchit.jud@mahidol.ac.th

1

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Outline of presentation

1) Homogeneity testing of PT test material and statistical evaluation
2) Stability testing of PT test material and statistical evaluation
3) Determination of assigned value and its standard uncertainty
4) Determination of criteria for evaluation of performance
5) Evaluation of laboratory performance

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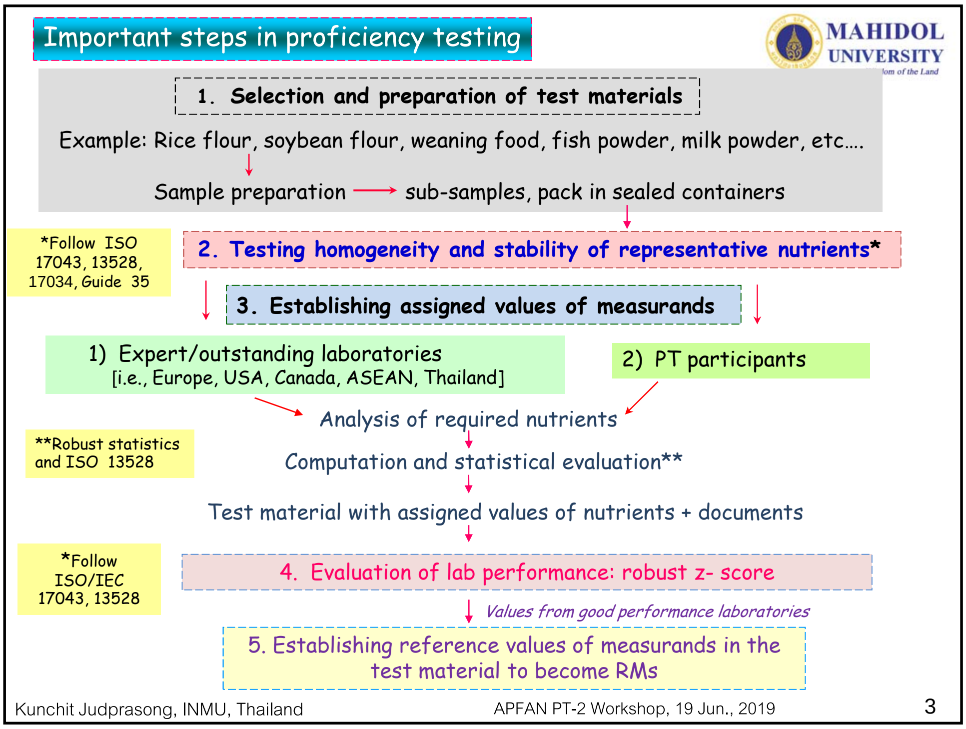
Important steps in proficiency testing
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1. Selection and preparation of test materials
Example: Rice flour, soybean flour, weaning food, fish powder, milk powder, etc....
Sample preparation → sub-samples, pack in sealed containers
*Follow ISO 17043, 13528, 17034, Guide 35
2. Testing homogeneity and stability of representative nutrients*
3. Establishing assigned values of measurands
1) Expert/outstanding laboratories [i.e., Europe, USA, Canada, ASEAN, Thailand]
2) PT participants
Analysis of required nutrients
**Robust statistics and ISO 13528
Computation and statistical evaluation**
Test material with assigned values of nutrients + documents
*Follow ISO/IEC 17043, 13528
4. Evaluation of lab performance: robust z- score
Values from good performance laboratories
5. Establishing reference values of measurands in the test material to become RMs
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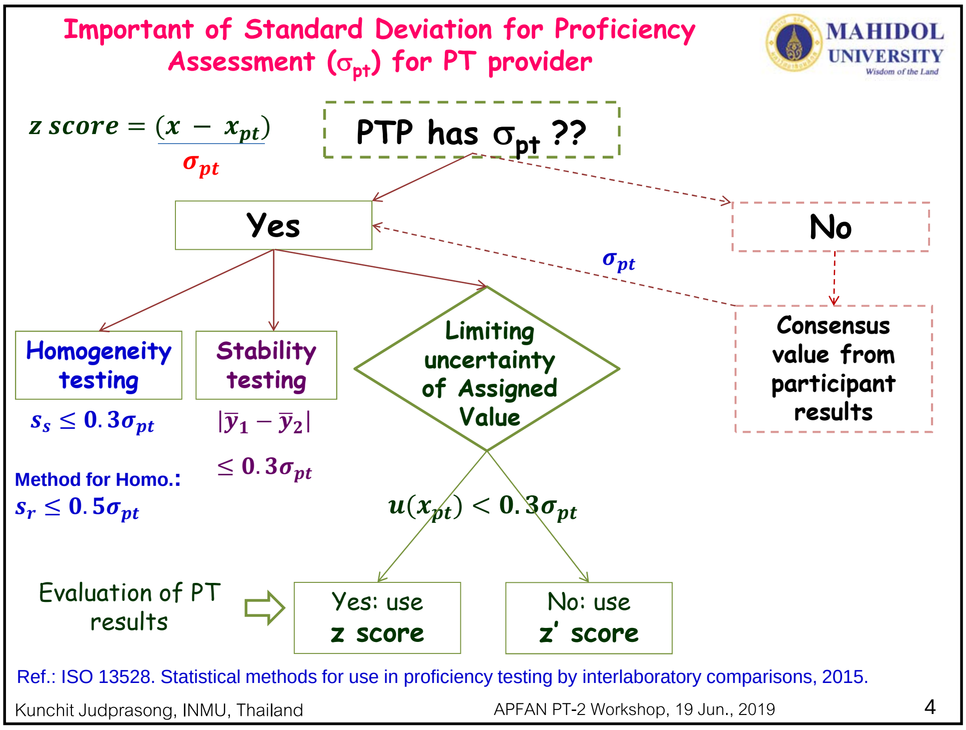
Important of Standard Deviation for Proficiency Assessment (σ_{pt}) for PT provider
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$z\ score = \frac{(x - x_{pt})}{\sigma_{pt}}$
PTP has σ_{pt} ??
Yes
No
σ_{pt}
Consensus value from participant results
Homogeneity testing
$s_s \leq 0.3\sigma_{pt}$
Method for Homo.:
$s_r \leq 0.5\sigma_{pt}$
Stability testing
$|\bar{y}_1 - \bar{y}_2| \leq 0.3\sigma_{pt}$
Limiting uncertainty of Assigned Value
$u(x_{pt}) < 0.3\sigma_{pt}$
Evaluation of PT results
Yes: use z score
No: use z' score
Ref.: ISO 13528. Statistical methods for use in proficiency testing by interlaboratory comparisons, 2015.
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ISO 13528: 2015 B.2 Assessment criteria for a homogeneity check

B.2.1 The following three checks should be used to assure that the homogeneity test data are valid for analysis:

a) Examine the results for each test portion in order of measurement to look for a trend (or drift) in analysis; if there is an apparent trend, take appropriate corrective action regarding the measurement method, or use caution in the interpretation of the results.

b) Examine the results for proficiency test item averages by production order; if there is **a serious trend** that causes the proficiency test item to exceed the criterion at B.2.2 ($s_s \leq 0{,}3\ \sigma_{pt}$) or otherwise prevents use of the proficiency test item, then (i) either assign individual values to each proficiency test item; or (ii) discard a subset of proficiency test items significantly affected and retest the remainder for sufficient homogeneity; or (iii) if the trend affects all proficiency test items, follow the provisions at B.2.4.

Key assessment criteria for a homogeneity testing (when known σ_{pt})

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Check trend (or drift) in analysis

⇩

Check trend (or drift) by production order

⇩

Test for a statistically significant difference between replicates, using Cochran's test

⇩

Checking for <u>between sample variation</u> (check homogeneity): **adequately homogeneous** $s_s \leq 0.3\sigma_{pt}$

⇩

If criteria is not met, apply expanded criteria approach: **sufficiently homogeneous** $s_s \leq \sqrt{c}\ , c = F_1\sigma_{all}^2 + F_2 s_{an}^2$

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

⇧

$u(x_{pt}) < 0{,}3\sigma_{pt}$

Include the s_s in the SDPA	Include s_s in the uncertainty of the assigned value and use z'	Discard the PT test item and repeat the preparation
$\sigma'_{pt} = \sqrt{\sigma_{pt}^2 + s_s^2}$	$z'_i = \frac{x_i - x_{pt}}{\sqrt{\sigma_{pt}^2 + u^2(x_{pt})}}$	

Ref.: ISO 13528. Statistical methods for use in proficiency testing by interlaboratory comparisons, 2015.

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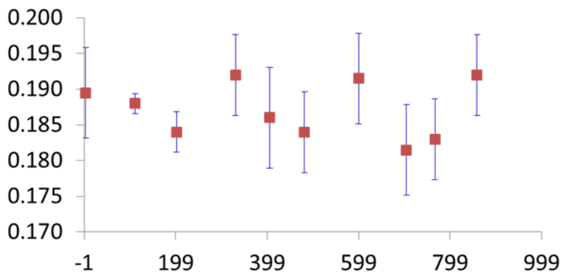
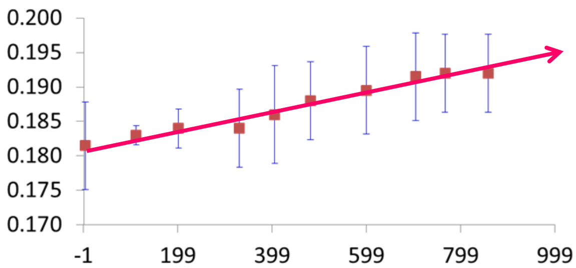
Examine the results for proficiency test item averages by production order

No trend



Have trend



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B.2 Assessment criteria for a homogeneity check

B.2.1 The following three checks should be used to assure that the homogeneity test data are valid for analysis:

c) Compare the difference between replicates (or range, if more than 2 replicates) and, if necessary, **test for a statistically significant difference between replicates, using Cochran's test (ISO 5725-2).** If the difference between replicates is large for any pair, review a technical explanation for the difference and if appropriate, remove the outlying group from the analysis or, if m>2 and the high variance is caused by a single outlier, remove the outlying point.

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Checking for within sample variation: Cochran's test

Duplicate analysis:
Cochran's maximum range test

Calculate the ratio: $\frac{D^2_{max}}{\sum D^2_i}$

where
D_{max} = *the maximum difference of the duplicates*
D_i = *difference of each pair of duplicates*

Triplicate analysis:
Cochran's maximum variance test

Calculate the ratio: $\frac{S^2_{max}}{\sum S^2_i}$

Where

S^2_{max} = *the maximum variance in the set*
S^2_i = *variance of each set of triplicate results*

- Compare the ratio to the critical value from Cochran Table (95% confidence).
- If the ratio is < critical value, there is no evidence of analytical outliers

- If any outlier is detected, results must be inspected closely to see any errors and appropriate action must be taken.
- An outlier set should not be rejected unless it is significant at 99% level or any permanent analytical errors are found.
- It should be excluded from ANOVA (calculation of MSW and MSB) unless there is a reason to the contrary.

When know σ_{pt}

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Statistical analysis: homogeneity testing

Checking for between sample variation (check homogeneity of the test materials) using

- ISO 13528:2015

$$s_s \leq 0.3\sigma_{pt} \longrightarrow \text{Adequately homogeneous}$$

- Expanded criteria or IUPAC (ISO 13528:2015; Thompson et al., 2006)

$$s^2_{sam} < c, \quad c = F_1\sigma^2_{all} + F_2 s^2_{an}$$

$$\text{or } s_s \leq \sqrt{c} \longrightarrow \text{Sufficiently homogeneous}$$

1) ISO 13528: 2015 Statistical methods for use in proficiency testing of interlaboratory comparisons.
2) Thompson M, Ellison SLR, Wood R. The International harmonised protocol for the proficiency testing of analytical chemistry laboratories (IUPAC Technical Report). Pure Appl Chem 78: 145-196, 2006.
3) ISO 5725-1994: Accuracy (trueness and precision) of measurement methods and results.

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B.2.5 If the criteria for sufficient homogeneity are not met, the proficiency testing provider shall consider adopting one of the following actions.

a) Include the between-sample standard deviation in the standard deviation for proficiency assessment, by calculating σ'_{pt} as in equation (B.3). Note this needs to be described fully to participants.

$$\sigma'_{pt} = \sqrt{\sigma_{pt}^2 + s_s^2} \qquad \text{(B.3)}$$

b) **Include s_s in the uncertainty of the assigned value and use z'** or δ_E' to assess performance (see 9.5);

c) When σ_{pt} is the robust standard deviation of participant results, then the inhomogeneity between proficiency test items is included in σ_{pt} and so the criterion for acceptability of homogeneity can be relaxed, with caution.

If none of a) to c) apply, discard the proficiency test item and repeat the preparation after correcting the cause of inhomogeneity.

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B.2.4 **When σ_{pt} is not known** in advance, for example when σ_{pt} is the robust standard deviation of participant results, the proficiency testing provider should choose other criteria for determining sufficient homogeneity. Such procedures could include:

a) check for statistically significant differences between proficiency test items using, for example, the **Analysis of Variance F test at α=0,05** ;

b) use information from **previous rounds** of the proficiency testing scheme to estimate σ_{pt} ;

c) use data from a precision experiment (such as a reproducibility standard deviation as described in ISO 5725-2);

d) **accept the risk of distributing proficiency test items** that are not sufficiently homogeneous, and check the criterion after the consensus σ_{pt} has been calculated.

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STABILITY TESTING

a. Classical stability study: i.e., vitamin B_1 in rice flour

- Store samples at storage conditions which represent conditions of the entire process - transportation and storage
- Re-analyse the suspected component in 5 randomly single samples at specified periods
- If the obtained values are in the range of mean $\pm$ 2SD derived from the homogeneity study or at 0 day storage; the component is stable

b. Isochronous stability study: i.e., minerals

- The sub-samples collected at different periods and kept at -40°C to -80°C
- The analytes in the sub-samples are measured at the same time
  ⟶ reduce measurement variation ⟶ smaller uncertainty

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a. Classical stability study: i.e., vitamin B_1 in rice flour

Day 0 Homogeneity	2 Week Transport stability	2 Month End of study
↓	↓	↓
Measurement	Measurement	Measurement

b. Isochronous stability study: i.e., minerals

Day 0 Homogeneity → **2 Week** Transport stability → **2 Month** End of study

↓

Kept at -40°C to -80°C

↓

Measurement in the same set

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ISO 13528: $|\bar{y}_1 - \bar{y}_2| \le 0.3\sigma_{pt}$

where $\bar{y}_1$ = mean value from homogeneity of test material

$\bar{y}_2$ = mean value from stability of each storage time

σ_{pt} = standard deviation for proficiency testing

Stability sample	Replicate 1	Replicate 2
164	0.191	0.198
732	0.19	0.196
$\|\bar{y}_1 - \bar{y}_2\| \le 0{,}3\sigma_{pt}$		
Overall average (y_1) =	0.19375	mg/kg
Mean from homogeneity (y_2) =	0.18715	mg/kg
Differece from homo. mean =	0.0066	mg/kg
σ_{PT} =	0.02807	mg/kg
Thus, 0.3 σ_{PT} =	0.00842	mg/kg
Summary:	Pass	

↓

Arsenic in chocolate is stable throughout the storage period of study.

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Determination of the assigned value &
Determination of criteria for evaluation of performance

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

x_{pt} →
- 7.3 Formulation
- 7.4 Certified reference material
- 7.5 Results from one laboratory
- 7.6 Consensus value from expert laboratories
- 7.7 Consensus value from participant results

σ_{pt} = standard deviation for proficiency assessment

- 8.2 By perception of experts
- 8.3 By experience from previous rounds of a proficiency testing scheme
- 8.4 By use of a general model
- 8.5 Using the repeatability and reproducibility standard deviations from a previous collaborative study of precision of a measurement method
- 8.6 From data obtained in the same round of a proficiency testing scheme

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1) By formulation

- ☺ addition of a known amount/concentration of a component to a base material
- ☺ base material must be effectively free from the component, or its original or residual component concentration is accurately known

Assigned value X is derived by calculation from the mass used.
Standard uncertainty u_x is estimated by combination of uncertainties (gravimetric and volumetric) using the approach in GUM

Drawbacks

- ☹ It may be difficult to mix the analyte homogeneously into the base medium (e.g., add liquid solution into solid powder
  (experience – spike mineral in egg and milk)*
- ☹ Added analyte is loosely bound or in different chemical form from the natural food sample

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2) Certified reference values

- Test material used: certified reference material
  - ☹ *matrix and an analyte concentration:* difficult to match with the test material !!!*

**appropriate CRMs are not usually available, restricts use of this method*

- ☹ *This approach is too expensive.*

Assigned value X is derived from certified reference value.
Standard uncertainty u_x is derived from uncertainty specified in the certificate.

- ☺ *Simple to implement and provides assigned values independent of the participants results*
- ☺ *Provides direct traceability and uncertainty*

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3) Results from one laboratory

- An assigned value can be determined by a single laboratory using a reference method, such as, for example, **a primary method**.
- This determination requires a series of tests to be carried out, in one laboratory, on proficiency test items and the CRM, using the same measurement method, and under repeatability conditions.

Assigned value $x_{pt} = x_{CRM} + \bar{d}$

Standard uncertainty $u_{x_{pt}} = \sqrt{u_{CRM}^2 + u_d^2}$

x_{pt} = the assigned value for the RM
x_{CRM} = the certified value for the CRM
$\bar{d}$ = the difference (x_{pt}-CRM) between the average results for the x_{pt} and the CRM

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4) Consensus values from expert laboratories

- obtained from a group of expert laboratories* using a protocol that specifies the numbers of proficiency test items
- Each expert laboratory is required to provide a standard uncertainty with their results.
- Where the expert laboratories report uncertainties with the results, the estimation of a value by consensus of results is a complex problem and **a wide variety of approaches has been suggested**, including, for example, weighted averages, un-weighted averages

Consensus from "expert laboratories": some considerations

- Criteria for expert laboratories; e.g., ISO accredited, good performance in previous PT studies, use high metrological methods
- Expert lab may analyse the test material at the same time as the participants

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4) Consensus values from expert laboratories (continued)

A. Assigned value: estimated as robust average from the results reported by the group of expert laboratories, using Algorithm A in Annex C (ISO 13528) . Other estimating methods may be used.

B. Standard uncertainty

$$u_X = \frac{1.25 \text{ x } s^*}{\sqrt{p}}$$

p = number of expert laboratories conducting a measurement
u_i = standard uncertainty of the measurement

When the expert laboratories do not report the value of standard uncertainty, the standard uncertainty of the assigned value shall be estimated from robust mean following the ISO 13528.

Estimation of assigned values:
Robust average (x^*) $\pm$ Robust SD (S^*) ;
Robust average (x^*) $\pm$ Uncertainty (u_x)

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5) Consensus value from participants results

- Most economical and effective means to obtain the assigned value
- Homogeneous food material is analysed by a number of participating laboratories

Choice of analytical methods

- Routine analytical methods
- Nationally or internationally accepted methods

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5) Consensus from participants in a round of PT scheme

Estimation of assigned values:

Algorithm A in Annex C of ISO 13528

Assigned value: Robust mean (x^*) ± robust SD (s^*),

Robust mean (x^*) ± uncertainty (u_x)

Standard uncertainty $$u_{x\,pt} = \frac{1.25 \text{ x } s^*}{\sqrt{p}}$$

s^* = the robust standard deviation of the results, calculated using Algorithm A in Annex C.' p = number of participants' reported data

Limitations:
- there may be no real consensus amongst the participants;
- number of participants should > 12
- the consensus may be biased by the general use of faulty methodology and this bias will not be reflected in the standard uncertainty of the assigned value

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Determination of the assigned value &

Determination of criteria for evaluation of performance

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

x_{pt}:
- 7.3 Formulation
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- 8.2 By perception of experts
- 8.3 By experience from previous rounds of a proficiency testing scheme
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8.0 Determination of criteria for evaluation of performance (ISO 13528: 2015)

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8.2 **By perception of experts**: set at a value that corresponds to the level of performance that a regulatory authority, accreditation body, or the technical experts of the proficiency testing provider believe is reasonable for participants.

8.3 **By experience from previous rounds of a proficiency testing scheme**:

- The review of previous rounds of a proficiency testing scheme should include consideration of performance that is achievable by competent participants.

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8. Determination of criteria for evaluation of performance

8.2 By experience from previous rounds of a proficiency testing scheme:

- When the criterion for evaluation of performance is based on consensus statistics from previous rounds of a proficiency testing scheme, **robust estimates** of the standard deviation should be used.

NOTE 1 **Algorithm S** (Annex C.4) provides a robust pooled standard deviation that is applicable when all previous rounds of a proficiency testing scheme under consideration have the same expected standard deviation or (if relative deviations are used for the assessment) the same relative standard deviation.

Example: calculation σ_{pt} using Algorithm S

Iteration	0	1		2		3		4		5		6		7	
$\psi = \eta x w*$			11.09		11.66		11.87		11.94		11.97		11.98		11.99
No.	RSD	w_i^*	(w_i^*)^2	w_i^*	(w_i^*)^2	w_i	(w_i^*)^2	w_i	(w_i^*)^2	w_i	(w_i^*)^2	w_i	(w_i^*)^2	w_i	(w_i^*)^2
59-12	4.60	4.60	21.1	4.60	21.1	4.60	21.1	4.60	21.1	4.60	21.1	4.60	21.1	4.60	21.1
61-11	4.94	4.94	24.4	4.94	24.4	4.94	24.4	4.94	24.4	4.94	24.4	4.94	24.4	4.94	24.4
60-12	5.22	5.22	27.3	5.22	27.3	5.22	27.3	5.22	27.3	5.22	27.3	5.22	27.3	5.22	27.3
59-11	6.70	6.70	44.8	6.70	44.8	6.70	44.8	6.70	44.8	6.70	44.8	6.70	44.8	6.70	44.8
60-11	7.68	7.68	59.0	7.68	59.0	7.68	59.0	7.68	59.0	7.68	59.0	7.68	59.0	7.68	59.0
61-12	9.68	9.68	93.7	9.68	93.7	9.68	93.7	9.68	93.7	9.68	93.7	9.68	93.7	9.68	93.7
59-21	9.98	9.98	99.7	9.98	99.7	9.98	99.7	9.98	99.7	9.98	99.7	9.98	99.7	9.98	99.7
59-22	10.39	10.39	107.9	10.39	107.9	10.39	107.9	10.39	107.9	10.39	107.9	10.39	107.9	10.39	107.9
60-22	12.24	11.09	122.9	11.66	135.9	11.87	140.8	11.94	142.6	11.97	143.3	11.98	143.6	11.99	143.7
60-21	13.54	11.09	122.9	11.66	135.9	11.87	140.8	11.94	142.6	11.97	143.3	11.98	143.6	11.99	143.7
w*	8.68	9.13		9.29		9.35		9.37		9.38		9.39		9.39	
$\sum(w_i^*)^2$			723.74		749.74		759.51		763.17		764.55		765.07		765.26

จำนวนข้อมูล (*n*)	10
Degree of freedom (*p*)	9
η	1.277
ψ	1.300
ζ	1.018

new $w^* = \zeta\sqrt{\Sigma(w_i^*)^2/p}$

Target RSD or SDPA (σ_{pt}) = 9.4 %

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Pooled relative standard deviation, RSD_{pool}

$$RSD_{pool} = \sqrt{\left(\frac{(n_1-1)\times RSD_1^2 + (n_2-1)\times RSD_2^2 + \ldots}{(n_1-1)+(n_2-1)+\ldots}\right)}$$

Year	PT round	Mean	SD	RSD	n		n-1	RSD^2	$(n-1)RSD^2$
2016	59-11	24.4	1.63	6.70	21		20	45	897
2016	59-12	86.3	3.97	4.60	20		19	21	402
2016	59-21	5.2	0.52	9.98	26		25	100	2492
2016	59-22	26.1	2.71	10.39	28		27	108	2914
2017	60-11	2.5	0.19	7.68	15		14	59	826
2017	60-12	86.8	4.53	5.22	15		14	27	382
2017	60-21	3.0	0.40	13.54	13		12	183	2201
2017	60-22	4.8	0.59	12.24	12		11	150	1649
2018	61-11	85.7	4.23	4.94	14		13	24	317
2018	61-12	4.9	0.47	9.68	15		14	94	1312
						Sum =	169		13391.8
							RSD_p =	8.90	%

V J Barwick and S L R Ellison. VAM Project 3.2.1 Development and Harmonisation of Measurement Uncertainty Principles. Part (d): Protocol for uncertainty validation data. January 2000. LGC/VAM/1998/088

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8. Determination of criteria for evaluation of performance

8.4 By use of a general model:

8.4.1 The value of the standard deviation for proficiency assessment can be derived from a general model for the reproducibility of the measurement method. This method has the advantage of objectivity and consistency across measurands, as well as being empirically based.

$$\sigma_R = \begin{cases} 0{,}22c & \text{when} \quad c < 1{,}2\times10^{-7} \\ 0{,}02c^{0{,}8495} & \text{when} \quad 1{,}2\times10^{-7} \le c \le 0{,}138 \\ 0{,}01c^{0{,}5} & \text{when} \quad c > 0{,}138 \end{cases}$$

where *c* is the mass fraction
(i.e. Concentration of vitamin B1 is 10 mg/100g, C = 10/100/1000 = 0.0001)

8. Determination of criteria for evaluation of performance

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8.5 Using the repeatability and reproducibility standard deviations from a previous collaborative study of precision of a measurement method

8.5.1 When the measurement method to be used in the proficiency testing scheme is standardized, and information on the repeatability (σ_r) and reproducibility (σ_R) of the method is available, the standard deviation for proficiency assessment (σ_{pt}) may be calculated using this information, as follows:

$$\sigma_{pt} = \sqrt{\sigma_R^2 - \sigma_r^2 \left(1 - 1/m\right)}$$

where *m* is the number of replicate measurements each participant is to perform in a round of the proficiency testing scheme.

This equation is derived from a basic random effects model from ISO 5725-2.

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8. Determination of criteria for evaluation of performance

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8.6 From data obtained in the same round of a proficiency testing scheme

8.6.3 The main advantages of this approach are **simplicity and conventional acceptance** due to successful use in many situations.

8.6.4 There are several disadvantages with this approach:

a) The value of σ_{pt} **may vary substantially from round to round**

b) Standard deviations **can be unreliable** when the number of participants in the PT scheme is small or when results from different methods are combined.

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Outline of presentation

1) Homogeneity testing of PT test material and statistical evaluation
2) Stability testing of PT test material and statistical evaluation
3) Determination of assigned value and its standard uncertainty
4) Determination of criteria for evaluation of performance
5) Evaluation of laboratory performance

Kunchit Judprasong, INMU, Thailand APFAN PT-2 Workshop, 19 Jun., 2019 35

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Laboratory performance by estimation of robust z-score

z score is used when $u(x_{pt}) < 0.3\sigma_{pt}$

Between laboratory variation: $z = \frac{(x_i - x_{pt})}{\sigma_{pt}}$

where x_i = average value of reported data from each participant

x_{pt} = the assigned value i.e. robust mean (x^*) obtained from ISO 13528 or median of the values obtained from participants

σ_{pt} = standard deviation for proficiency assessment i.e. robust standard deviation (s^*) obtained from ISO 13528 or predicted SD from previous round of PTs

Interpretation

$|z| \leq 2$ Satisfactory (acceptable) result
$2 < |z| < 3$ Questionable (warning) result
$|z| \geq 3$ Unsatisfactory (unacceptable) result

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Laboratory performance by estimation of z' -score

z' score will be used when $u(x_{pt}) > 0.3\sigma_{pt}$

Between laboratory variation: $$z' = \frac{(x_i - x_{pt})}{\sqrt{\sigma_{pt}^2 + u_{x_{pt}}^2}}$$

where x_i = average value of reported data from each participant

x_{pt} = the assigned value i.e. robust mean (x^*) or median of the values

σ_{pt} = standard deviation for proficiency assessment i.e. robust standard deviation (s^*) or predicted SD from previous round of PTs

$u_{x_{pt}}$ = the standard uncertainty of the assigned value x_{pt}

Interpretation

$|z'| \leq 2$ Satisfactory (acceptable) result

$2 < |z'| < 3$ Questionable (warning) result

$|z'| \geq 3$ Unsatisfactory (unacceptable) result

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Laboratory performance: Zeta score

- Zeta scores can be useful when an objective for the proficiency testing scheme is to evaluate a participant's ability to have results be close to the assigned value within their claimed uncertainty.

$$\zeta_i = \frac{(x_i - x_{pt})}{\sqrt{u_{x_i}^2 + u_{x_{pt}}^2}}$$

where x_i = value of reported data from each participating laboratory;

x_{pt} = the assigned value i.e. robust mean (x^*), median, reference value;

u_{x_i} = the participant's estimate of the standard uncertainty of its result x_i;

$u_{x_{pt}}$ = the standard uncertainty of the assigned value x_{pt}

- ζ scores above 2 or below -2 may be caused by systematically biased methods or by a poor estimation of the measurement uncertainty by the participant.
- ζ scores therefore provide a rigorous assessment of the complete result submitted by the participant.

E_n score

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- En scores can be useful when an objective for the proficiency testing scheme is to evaluate a participant's ability to have results close to the assigned value within their claimed expanded uncertainty.

$$E_n = \frac{(x_i - x_{pt})}{\sqrt{U_{x_i}^2 + U_{x_{pt}}^2}}$$

where x_i = value of reported data from each participating laboratory;

x_{pt} = the assigned value i.e. robust mean (x^*), median, reference value;

U_{x_i} = the participant's estimate of the expanded uncertainty of its result x_i;

$U_{x_{pt}}$ = the expanded uncertainty of the assigned value x_{pt}

Note: Direct combination of expanded uncertainties is not consistent with the requirement of ISO/IEC Guide 98-3 and is not equivalent to the calculation of a combined expanded uncertainty unless both the coverage factors and the effective degrees of freedom are identical for $U_{(xi)}$ and $U_{(xpt)}$.

- $-1.0 < En < 1.0$ should be taken as an indicator of successful performance

Summary statistic in proficiency testing

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Statistics in ISO 13528: 2015

1. Testing homogeneity of measurand →
- Cochran's test
- $s_s \leq 0.3\sigma_{pt}$
- $s_s \leq \sqrt{c}$ $\quad c = F_1\sigma_{all}^2 + F_2 s_{an}^2$

2. Testing stability of measurand →
- $|\bar{y}_1 - \bar{y}_2| \leq 0.3\sigma_{pt}$

3. Establishing assigned values of measurands →
- Robust statistic:
  - x*, median, reference value
  - s*, SDp, NIQR

4. Evaluation of lab performance →
- $z = \frac{(x_i - x_{pt})}{\sigma_{pt}}$
- $z' = \frac{(x_i - x_{pt})}{\sqrt{\sigma_{pt}^2 + u_{x_{pt}}^2}}$
- $\zeta_i = \frac{(x_i - x_{pt})}{\sqrt{u_{x_i}^2 + u_{x_{pt}}^2}}$
- $E_n = \frac{(x_i - x_{pt})}{\sqrt{U_{x_i}^2 + U_{x_{pt}}^2}}$