



PROFICIENCY TESTING PLAN

ZZB 2018/1 – Strength and durability of hardened concrete
(ZZB 12390, 480-11, 731322, 731324, 731326)

Proficiency Testing Provider at the SZK FAST
Veveří 95, 602 00 Brno
Czech Republic

www.szok.fce.vutbr.cz

Coordinator
Supervisor
Approved for PT Provider

doc. Ing. Tomáš Vymazal, Ph.D.
Ing. Petr Misák, Ph.D.
doc. Ing. Tomáš Vymazal, Ph.D.

Approved: 04/08/2017
Approved: 04/08/2017
Approved: 04/08/2017

Contents

1	Basic Information about the Proficiency Testing Program	2
2	Implementation of the Proficiency Testing Program	2
2.1	Specifications and Characteristics	2
2.2	Ensuring Homogeneity and Stability	4
2.3	Instructions for the Elimination of Main Error Sources	4
2.4	PTP Schedule	5
3	Procedures used in the Statistical Analysis of Laboratory Results	5
3.1	The Numerical Procedure for Determining Outliers	5
3.1.1	Cochran's test	5
3.1.2	Grubbs' test – One Outlying Observation	6
3.2	Mandel's Statistics	6
3.2.1	Interlaboratory Consistency Statistic h	6
3.2.2	Intralaboratory Consistency Statistic k	6
3.3	Calculation of Variances Estimates	7
3.3.1	Repeatability Variance	7
3.3.2	Interlaboratory Variance	7
3.3.3	Reproducibility Variance	7
3.4	Repeatability and Reproducibility	7
3.5	Assigned Values	8
3.6	Calculation of Performance Statistics	9
4	Certificate of Participation and the Final Report on the Results of Interlaboratory Comparison	9
5	Safeguards for Confidentiality	9
6	Related Documents	10

1 Basic Information about the Proficiency Testing Program

The aim of the Proficiency Testing Program (PTP) is to compare and evaluate the results of tests conducted on hardened concrete in compliance with selected parts of EN 12390 ([1], [2] and [2]), EN 480-11 [3], ČSN 731322 [4], ČSN 731324 [5] and ČSN 731326, method A and C [6].

The program strives to provide objective information about the measuring skills of PTP participants. The basic criterion for participation is timely registration for the program, and the prerequisites for obtaining the Certificate of Participation and the Final Report on the Results of Interlaboratory Comparison are timely payment of the fee and adherence to the schedule.

Important dates:

Registration deadline:	31/01/2018
Distribution of samples:	09 – 13/04/2018
Realization/initiation of testing:	23/04/2018
Results sent to the organizer:	18/05/2018
Evaluation/presentation of Certificate of Participation:	30/06/2018

2 Implementation of the Proficiency Testing Program

2.1 Specifications and Characteristics

Testing laboratories and other institutions interested can register for the PTP. The minimum number of participants is 5. If the number of participants is close to the minimum, the coordinator will consider the evaluation of PTP results using Horn's procedure to determine the assigned value and measurement uncertainty. The maximum number of participants is 30. If the minimum number of participants is not reached, the PT Provider reserves the right to cancel the PTP. This takes place according to Chapter 3 of the "Cancellation and Complaint Proceedings" instructions [7] available on www.szk.fce.vutbr.cz/en.

Parts of the PT program:

1. EN 12390-3 [1]

- Characteristics: Compressive strength
- Unit: N/mm²
- Specification according to EN 206 [8]: C 30/37
- Testing specimens: 150 × 150 × 150 mm
- Number of observations: 3

2. EN 12390-7 [2]

- Characteristics: Weight Density
- Unit: kg/m³
- Specification according to EN 206 [8]: Ordinary concrete
- Testing specimens: 150 × 150 × 150 mm
- Number of observations: 3
- Instructions:
 - (a) Perform with the specimens prior to compressive strength testing according to EN 12390-3 [1].
 - (b) During the determination of the observed property of hardened concrete, prefer the method according to chap. 5.3. EN 12390-7 [2].

3. EN 12390-8 [9]

- Characteristics: Depth of penetration of water under pressure
- Unit: mm

- Specification according to EN 206 [8]: XF2
- Testing specimens: 150 x 150 x 150 mm
- Number of observations: 3
- Instructions:
 - (a) Perform with any side face of the cubes, i.e. not the top nor bottom.
 - (b) The sides of the cubes have been treated according to article 6.1 of EN 12390-8 [9].

4. EN 480-11 [3]

- Characteristics: Total air content; A300; L
- Unit: %, %, mm
- Specification according to EN 206 [8]: 3 – 7
- Testing specimens: 150 x 150 x 150 mm
- Number of observations: 2 (4 specimens)
- Instructions:
 - (a) The samples will be prepared by the participants themselves according to article 6 in EN 480-11[3].
 - (b) The mixture formula will be provided by the organiser.

5. ČSN 731322 [4]

- Characteristics: Freeze-thaw resistance
- Unit: -
- Specification according to EN 206 [8]: C30/37
- Testing specimens: 100 x 100 x 400 mm
- Number of observations: 3+3 (50 cycles)
- Instructions:
 - (a) For the determination of the observed property of hardened concrete, 6 beam specimens sized 100 x 100 x 400 mm will be provided.
 - (b) Record the results after 50 cycles.
 - (c) Freezing and thawing takes place in cycles for concretes exposed to temperatures as low as -20°C.
 - (d) Frost resistance coefficient is the ratio of the arithmetic average of flexural strength of the frozen beams to the arithmetic average of flexural strength of reference beams.

6. ČSN 731324 [5]

- Characteristics: R_o
- Unit: %
- Specification according to EN 206 [8]: -
- Testing specimens: 7.07 x 7.07 x 7.07 mm
- Number of observations: 3/6
- Instructions: 3 or 6 cube specimens sized 7.07mm \pm 2% (depending on the number of participants) will be supplied for the determination of the observed property of hardened concrete.

7. ČSN 731326, method A [6]

- Characteristics: Freeze-thaw resistance
- Unit: g/m²
- Specification according to EN 206 [8]: XF2
- Testing specimens: 150 x 150 x 150 mm
- Number of observations: 3 x 100 cycles

- Instructions: Three cube specimens sized 150 mm will be provided by the organiser for the determination of the observed property of hardened concrete.

8. ČSN 731326, method C [6]

- Characteristics: Freeze-thaw resistance
- Unit: g/m^2
- Specification according to EN 206 [8]: XF2
- Testing specimens: Cylinder 50 x 150mm
- Number of observations: 3 x 100 cycles
- Instructions: 3 cylinder specimens (offcuts) of 150 mm in diameter and 50 mm height will be supplied for the determination of the observed property of hardened concrete.

9. EN 12390-9 [10]

- Characteristics: Freeze-thaw resistance – Scaling
- Unit: g/m^2
- Specification according to EN 206 [8]: XF2
- Testing specimens: part of 150 x 150 x 150 mm
- Number of observations: 3
- Instructions: 3 cuboid specimens (offcuts) of 150 mm in width and 50 mm height will be supplied for the determination of the observed property of hardened concrete.

2.2 Ensuring Homogeneity and Stability

PT Provider employees and any suppliers they may utilize are aware of the significance of the homogeneity and stability of test specimens for the results of the Proficiency Testing Program. The homogeneity and stability of specimens is ensured in the following ways:

1. the material used for the production of samples is always taken from the same production and is of the same production date; and/or
2. by dividing the specimens produced in different batches in order to ensure specimen homogeneity during testing of physical-mechanical and durability properties,
3. using a single type of mold-release preparation,
4. using a single type of molds from the same material for one type of test,
5. storing all specimens together under identical conditions,
6. checking all specimens before dispatching to participants.

2.3 Instructions for the Elimination of Main Error Sources

PTP participants have the obligation:

- to store and transport the test specimens according to EN 12390-2 [11],
- to handle the proficiency testing materials in the same way they handle the majority of routinely tested samples,
- to follow the instructions of the PT Provider employee responsible for the PTP, especially regarding the type of testing carried out, the number of result determinations and the PT schedule,
- to state measurement uncertainties in accordance with their documented procedures, including the corresponding expansion coefficient. Participants will use expansion coefficient 2, which approximately represents the 95 % reliability level, unless stated otherwise,
- to adhere to the rules and principles of ethical conduct, as well as to regulations governing health and safety at work and fire safety, and to use exclusively electrical devices and facilities with a valid inspection report,
- to send the test results obtained during proficiency testing, including measurement uncertainties, to the PT Provider by the set deadline (see part 1).

2.4 PTP Schedule

All other information, forms and records not included in this document are accessible in updated form at www.szk.fce.vutbr.cz/en.

3 Procedures used in the Statistical Analysis of Laboratory Results

To describe the accuracy of measuring methods, the terms trueness and precision are used. Trueness refers to the closeness to congruity between the arithmetic mean of a high number of test results and a real or accepted reference value. Precision means the closeness to congruity between test results. The necessity to consider precision is based on the fact that tests generally do not yield the same results even though they are supposed to be carried out on the same material and under the same conditions. This is caused by accidental errors that are impossible to avoid. These errors represent an integral part of every testing procedure and we are unable to control them fully. The comparative analysis of laboratory data does not focus on assessing the trueness of test results, but first and foremost on their precision. Results are thus compared with one another and not with any reference value or real value.

The basis of the statistical analysis is a critical data assessment complying with ISO 5725-2 [12], i.e. the determination of dubious and outlying values, and other irregularities. This assessment is carried out using mainly Grubbs' and Cochran's tests (numerical evaluation) as well as Mandel's statistics (graphical evaluation). Other observed statistical parameters are interlaboratory dispersion, repeatability dispersion, reproducibility dispersion and related characteristics of repeatability and reproducibility. The outcome of PTP is to assess the performance of participating laboratories in compliance with EN ISO/IEC 17043 [13], consisting of the determination of relative values and their uncertainties and a final comparison with the test results of PTP participants.

A prerequisite for using these methods is the unimodal probability distribution of measured data. Furthermore, p will stand for the number of participating laboratories marked by the index A prerequisite for using these methods is the unimodal probability distribution of measured data. Furthermore, p will stand for the number of participating laboratories marked by the index $i = 1, \dots, p$, each of which carried out n number of tests., each of which carried out n number of tests.

3.1 The Numerical Procedure for Determining Outliers

To determine outliers, two basic statistical tests are used. One of them is Cochran's C test, which tests interlaboratory variabilities (in cases when the number of measurements of one quantity in one laboratory > 2) and is used first. If this test marks one participant's results as outlying, the laboratory is excluded and the test repeated. The second test (Grubbs' test) is first and foremost a test of interlaboratory variability and we can also employ it if Cochran's test raises the suspicion that only one of the test results is to blame for the high interlaboratory dispersion. Both tests assume a balanced experiment, i.e. the number of tests at one laboratory for the determination of one quantity must be constant.

When determining divergent or outlying values, three situations can occur:

- If the test statistic is within or equal to 5% of the critical value, the tested entity is considered to be *correct*;
- If the test statistic diverges from the critical value by more than 5%, but is within or equal to 1% of the critical value, the tested entity is considered to be *divergent*;
- If the test statistic diverges from the critical value by more than 1%, the tested entity is considered to be *outlying*.

3.1.1 Cochran's test

The Cochran's C statistic is given by the equation:

$$C = \frac{s_{max}^2}{\sum_{i=1}^p s_i^2} \quad (1)$$

where s_{max} is the highest sample standard deviation, s_i are sample standard deviations determined according to the results from all laboratories and p means the number of laboratories participating in the PT program.

The sample standard deviation is determined from the equation

$$s_i = \sqrt{\frac{1}{n_i - 1} \sum_{k=1}^{n_i} (y_k - \bar{y})^2}, \quad (2)$$

where n_i is the number of test results from the determination of one quantity in i -th laboratory, y_k is the k -th value and \bar{y} is the average value measured in the i -th laboratory. If only two results were measured for the relevant quantity, we can use the simplified equation:

$$s_i = \frac{|y_1 - y_2|}{\sqrt{2}}. \quad (3)$$

3.1.2 Grubbs' test – One Outlying Observation

From the given set of x_i data for $i = 1, 2, \dots, p$, ordered upward according to size, Grubbs' statistic G_p is calculated in order to use Grubbs' test to determine whether the largest observation is an outlier:

$$G_p = \frac{x_p - \bar{x}}{s}, \quad (4)$$

whereby \bar{x} is the arithmetic mean of the observed feature. The observed feature can be the average value of the quantity determined within the laboratory. Furthermore, s is a sample standard deviation of the observed feature, which in this case is a standard deviation calculated for all the laboratories.

For significance testing of the smallest observation the test statistic is calculated:

$$G_p = \frac{\bar{x} - x_p}{s}. \quad (5)$$

3.2 Mandel's Statistics

In order to determine data consistency, two values called Mandel's h and k statistics were used. These indicators are commonly used for the graphical evaluation of laboratories in a similar way to a description of variability.

3.2.1 Interlaboratory Consistency Statistic h

For each laboratory, the interlaboratory consistency statistic h was evaluated according to the formula

$$h_i = \frac{\bar{y}_i - \bar{\bar{y}}}{\sqrt{\frac{1}{p-1} \sum_{i=1}^p (\bar{y}_i - \bar{\bar{y}})^2}}, \quad (6)$$

where \bar{y}_i is the average value for the i -th laboratory, $\bar{\bar{y}}$ is the arithmetic mean of all values and p is the number of laboratories. The values of the h_i statistics were plotted on graphs.

3.2.2 Intralaboratory Consistency Statistic k

The intralaboratory consistency statistic k is calculated from the equation

$$k_i = \frac{s_i \sqrt{p}}{\sqrt{\sum_{i=1}^p s_i^2}}, \quad (7)$$

where s_i is a sample standard deviation of values measured at the i -th laboratory. Just as with h statistics, the k values are plotted on graphs.

Study of the graphs displaying h and k values may indicate that certain laboratories show a significantly different ordering of results than other studied laboratories. This is caused by a permanently large and/or permanently small dispersion of results or extreme averages of results across all levels.

3.3 Calculation of Variances Estimates

After the elimination of outliers (of laboratories), we can proceed to the calculation of basic variability characteristics, i.e. repeatability dispersion, interlaboratory dispersion and reproducibility dispersion. These characteristics are stated in the form of standard deviations, i.e. after extracting the root. It is advantageous when the variability characteristics and the observed quantity are of the same physical dimensions.

3.3.1 Repeatability Variance

$$s_r^2 = \frac{\sum_{i=1}^p (n_i - 1) s_i^2}{\sum_{i=1}^p (n_i - 1)} \quad (8)$$

3.3.2 Interlaboratory Variance

$$s_L^2 = \frac{s_d^2 - s_r^2}{\bar{n}}, \quad (9)$$

where

$$s_d^2 = \frac{1}{p-1} \sum_{i=1}^p n_i (\bar{y}_i - \bar{y})^2 \quad (10)$$

and

$$\bar{n} = \frac{1}{p-1} \left[\sum_{i=1}^p n_i - \frac{\sum_{i=1}^p n_i^2}{\sum_{i=1}^p n_i} \right]. \quad (11)$$

3.3.3 Reproducibility Variance

$$s_R^2 = s_r^2 + s_L^2, \quad (12)$$

where s_r^2 is repeatability variance and s_L^2 is interlaboratory variance.

3.4 Repeatability and Reproducibility

Repeatability expresses the fact that the difference between two test results from the same sample from tests carried out by the same person at the same facility and within the shortest time interval possible will not exceed the repeatability value r on average more than once in 20 cases if the method is employed in the common and correct manner.

The repeatability value is expressed by the relation

$$r = 2, 8 s_r, \quad (13)$$

where $s_r = \sqrt{s_r^2}$ stands for the standard deviation of repeatability.

Reproducibility expresses the fact that the reproducibility value R for test results from one sample obtained in the shortest time interval possible by two persons who used their own devices will not differ on average more than once in 20 cases if the method is employed in the common and correct manner.

The reproducibility value is expressed by the relation

$$R = 2, 8 s_R, \quad (14)$$

where $s_R = \sqrt{s_R^2}$ stands for the standard deviation of reproducibility.

3.5 Assigned Values

The PT Provider will ensure the determination of assigned value X and its uncertainty for every PTP. Assigned values are always only imparted to PTP participants after they have submitted their PTP results so that they cannot obtain any benefit from the premature revelation of the values.

The assigned values are determined by the PT Provider as consensual values derived from the results of participants in compliance with Appendix B of EN ISO/IEC 17043 [13] using the statistical methods described in ISO 13528 [14] and ISO 5725-5 [15]. The assigned value X is therefore determined as a robust estimate of the average value x^* (the A algorithm mentioned in [14] and [15]):

Initial values x^* and s^* (robust standard deviation) are calculated as

$$x^* = \text{median } x_i, \quad (15)$$

$$s^* = 1,483 \cdot \text{median } |x_i - x^*|, \quad (16)$$

where $i = 1, \dots, p$. The values of x^* and s^* are then processed as follows. First, $\varphi = 1,5 \cdot s^*$ is computed. For every x_i ($i = 1, \dots, p$) value, the following is calculated

$$x_i^* = \begin{cases} x^* - \varphi & \text{if } x_i < x^* - \varphi, \\ x^* + \varphi & \text{if } x_i > x^* + \varphi, \\ x_i & \text{in other cases.} \end{cases} \quad (17)$$

New values of x^* and s^* are calculated from the following equations

$$x^* = \sum_{i=1}^p \frac{x_i^*}{p}, \quad (18)$$

$$s^* = 1,134 \cdot \sqrt{\sum_{i=1}^p \frac{(x_i^* - x^*)^2}{p-1}}. \quad (19)$$

Robust estimates are derived by iteration until the estimate changes between calculations become small.

The standard uncertainty u_X of an assigned value determined in this manner is calculated from the relation

$$u_X = 1,25 \cdot \frac{s^*}{\sqrt{p}}. \quad (20)$$

In the case of a small number of PTP participants, the PT Provider sets the assigned values as consensual values obtained from expert participants who have proven their competence to determine the measured quantity that is the subject of testing.

Furthermore, if the number of participants is small ($4 \leq p \leq 20$), the PT Provider can consider determining the relative values by using what is called **Horn's method**. This method consists in the determination of so-called pivots used as a basis for estimating location and variability. First, the assessed data are ordered upwards. The low pivot is then determined from the equation

$$x_D = x_{(H)}, \quad (21)$$

where H is an ordinal index given by the equation $H = \frac{\text{int}(\frac{p+1}{2})}{2}$ or $H = \frac{\text{int}(\frac{p+1}{2} + 1)}{2}$.

The upper pivot is then determined from the equation

$$x_H = x_{p+1-H}. \quad (22)$$

Using Horn's method, the assigned value is determined as a location estimate, i.e. as the so-called pivot half sum:

$$x^* = \frac{x_D + x_H}{2}. \quad (23)$$

The variability estimate is determined as the so-called pivot range

$$R_L = x_H - x_D \quad (24)$$

and the uncertainty of an assigned value calculated in this way is determined as a 95% interval estimate of the mean value

$$u_X = R_L \cdot t_{L;0,95}(p), \quad (25)$$

where $t_{L;0,95}(p)$ is the $(1 - \alpha)$ quantile of the T_L probability distribution with p degrees of freedom.

3.6 Calculation of Performance Statistics

Proficiency test results often need to be transformed into performance statistics in order to aid interpretation and to allow comparison with defined objectives. The aim is to express the divergence from the assigned value in a way that enables its comparison with performance criteria. In compliance with the EN ISO/IEC 17043 standard [13], the performance of participating laboratories is evaluated according to the so-called z-score and ζ -score (zeta-score).

For every non-outlying laboratory (participant), the z-score is calculated according to the equation

$$z_i = \frac{|\bar{x}_i - x^*|}{s^*}. \quad (26)$$

ζ -score is calculated using the equation

$$\zeta_i = \frac{|\bar{x}_i - x^*|}{\sqrt{u_i^2 + u_X^2}}, \quad (27)$$

where u_i is a combined standard uncertainty of the i -th laboratory. Combined standard measurement uncertainties can be arrived at by dividing the extended uncertainty U by the extension coefficient k , which for normal probability division has the value $k = 2$. If the participant does not state the extended measurement uncertainty in their test result protocol, it is impossible to determine the ζ -score. For more about measurement uncertainties see document [16].

The following scales are applied for the z-score and ζ -score (to simplify the matter, only the z-score is shown):

$$z\text{-score} = \begin{cases} |z| \leq 2 & \text{shows that the laboratory performance is **satisfactory** and generates no signal;} \\ 2 \leq |z| \leq 3 & \text{shows that the laboratory performance is **questionable** and generates an action signal;} \\ 3 \leq |z| & \text{shows that the laboratory performance is **unsatisfactory** and generates an action signal.} \end{cases} \quad (28)$$

4 Certificate of Participation and the Final Report on the Results of Interlaboratory Comparison

The PT Provider gives expert commentary on participant efficiency evaluation in the Final Report as part of training courses the PT Provider organises. The Final Report preserves the anonymity of the PTP participants. Each participant, or the participant's test results, is represented by an ID number. The Certificate of Participation in the PT programme is part of the Final Report. The Certificate is unique to each participant and includes the participant's ID number.

5 Safeguards for Confidentiality

The identity of PTP participants is confidential and only known to persons/subjects involved with the PTP. All participant information is considered confidential by the PT Provider. The participant may renounce this confidentiality for the purposes of discussion and mutual assistance until the PTP results are obtained. The PT Provider reveals the proficiency testing results to no third party with the sole exception of a written request by a regulatory authority submitted prior to the commencement of the PTP and which has been granted a written consent by the PTP participants.

6 Related Documents

- Appendix 1 – Measurement Record – www.szk.fce.vutbr.cz/en
- Quality Handbook of the PT Provider at the SZK FAST
- Cancellation and Complaint Proceedings available at www.szk.fce.vutbr.cz [7]
- MPA 20 – 01 - . . . for application of EN ISO/IEC 17043 Concordance Assessment – General Requirements for Proficiency Testing in the Accreditation System of the Czech Republic.

References

- [1] EN 12390-3. *Testing hardened concrete - Part 3: Compressive strength of test specimens*. 2009.
- [2] EN 12390-7. *Testing hardened concrete - Part 7: Density of hardened concrete*. 2009.
- [3] EN 480-11. *Admixtures for concrete, mortar and grout - Test methods - Part 11: Determination of air void characteristics in hardened concrete*. 2006.
- [4] ČSN 73 1322. *Determination of frost resistance of concrete*. 2003.
- [5] ČSN 73 1324. *Determination of grindability of concrete*. 2003.
- [6] ČSN 73 1326. *Resistance of cement concrete surface to water and defrosting chemicals*. 2003.
- [7] *Cancellation and Complaint Proceedings – available at www.szk.fce.vutbr.cz/en*.
- [8] EN 206. *Concrete - Specification, performance, production and conformity*. 2014.
- [9] EN 12390-8. *Testing hardened concrete - Part 8: Depth of penetration of water under pressure*. 2009.
- [10] CEN/TS 12390-9. *Testing hardened concrete - Part 9: Freeze-thaw resistance - Scaling*. 2007.
- [11] EN 12390-2. *Testing hardened concrete - Part 2: Making and curing specimens for strength tests*. 2009.
- [12] ISO 5725-2. *Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*. 1997.
- [13] EN ISO/IEC 17043. *Conformity assessment - General requirements for proficiency testing*. 2010.
- [14] ISO 13 528. *Statistical methods for use in proficiency testing by interlaboratory comparisons*. 2005.
- [15] ISO 5725-5. *Accuracy (trueness and precision) of measurement methods and results - Part 5: Alternative methods for the determination of the precision of a standard measurement method*. 1999.
- [16] EA 4/02. *Vyjadřování nejistot měření při kalibracích*. 2000.