

PTP Plan

ZK 2017/1

Aggregates testing

(ZK 933, 1097, 1367, 137, 721179)

Proficiency Testing Provider at the SZK FAST
Brno University of Technology
Department of Building Testing
Faculty of Civil Engineering
Veveří 95
602 00 Brno
Czech Republic

Coordinator: 13/07/2016 Doc. Ing. Tomáš Vymazal, Ph.D.

Supervisor: 13/07/2016 Ing. Petr Misák, Ph.D.

Approved for PT Provider: 13/07/2016 Doc. Ing. Tomáš Vymazal, Ph.D.

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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 aggregate in compliance with selected parts of EN 933, EN 1097, EN 1367, TP137 and ČSN 72 1179 standards.

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:** 28/02/2017
- **Distribution of specimens:** 10 - 13/04/2017
- **Realization/initiation of testing:** 24/04/2017
- **Results sent to the organizer:** 31/05/2017
- **Evaluation/presentation of Certificate of Participation:** 30/06/2017

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 relative values and the target standard deviation. In that case, the participants will be told before PTP materials are distributed. 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 available on www.szk.fce.vutbr.cz/en.

The program usually focuses on the characteristics of aggregates – see **Table 1**

Table 1: Material specifications and characteristics

No.	Specification	Property	Range of observed parameters	Units	Material	Number of determinations
1	EN 933-1 ¹⁾	Particle size distribution	max G _c 80/20	%	Fine aggregate (0/4)	3 ²⁾
2	EN 933-3	Flakiness index (FI)	max FI ₃₅	%	Coarse aggregate	3 ³⁾
3	EN 933-4	Shape index (SI)	max SI ₄₀	%	Coarse aggregate	3 ³⁾
4	EN 933-5	Percentage of crushed and broken surfaces in coarse aggregate particles (C _{tc} , C _c , C _{tr} , C _r)	-	%	Coarse aggregate	3 ³⁾
5	EN 933-8 ⁸⁾	Sand equivalent test (SE)	-	-	Fine aggregate (0/4)	3 ²⁾
6	EN 933-9	MB value	-	g/kg	Fine aggregate (0/4)	3 ²⁾
7	EN 933-10	Grading of filler aggregates	-	%	Filer	3 ²⁾
8	EN 1097-1	Determination of the resistance to wear (M _{DE})	max M _{DE} 20	-	Coarse aggregate	1 ⁴⁾
9	EN 1097-2, Chapt. 5	Determination of resistance to fragmentation (LA)	max LA ₃₀	-	Coarse aggregate	1 ⁵⁾
10	EN 1097-2, Chapt.. 6	Determination of resistance to fragmentation (SZ)	max SZ ₂₆	%	Coarse aggregate	1 ⁶⁾
11	EN 1097-3	Determination of loose bulk density and voids (ρ _b , V)	-	Mg/m ³ , %	Fine aggregate	3 ²⁾
12	EN 1097-5	Water content (W)	-	%	Fine aggregate	3 ²⁾
13	EN 1097-6, Chapt.. 8	Determination of particle density and water absorption (ρ _a , WA ₂₄)	-	Mg/m ³ , %	Coarse aggregate	3 ²⁾
14	EN 1097-7	Determination of the particle density of filer (ρ _f)	-	Mg/m ³	Filer	1 ²⁾
15	EN 1367-1	Resistance to freezing and thawing (F)	max F ₄	%	Coarse aggregate	1 ²⁾
16	EN 1367-2	Magnesium sulfate test (MS)	max MS ₃₅	%	Coarse aggregate	1 ⁷⁾
17	EN 1367-3	Weight loss (M ₁)	-	%	Coarse aggregate	3
		Strength loss (S _{LA})	-	%	Coarse aggregate	3
18	TP 137 Appendix 1 a 2	Reactivity of aggregates in connection with alkalis	-	%	Coarse aggregate	1 ²⁾
19	ČSN 721179 – Appendix B	Reactivity of aggregates in connection with alkalis	-	%	Coarse aggregate	1 ²⁾

- 1) Perform using sieves with aperture size 4, 2, 1, 0,5 0.25, 0.125 a 0.063 mm – dry sample without washing.
- 2) A collective sample will be prepared from which you prepare 3 sub-specimens.
- 3) Perform 3 times on the same sample
- 4) Two unwashed sub-specimens weighing (500±2g) will be prepared in accordance with clause 6 EN 1097-1

- 5) One unwashed specimen weighing $5000\pm 5g$ will be prepared in accordance with clause 5.2 EN 1097-2
- 6) One unwashed specimen will be prepared in accordance with clause 6.2 EN 1097-2
- 7) Two unwashed sub-specimens will be weighing 500g will be prepared in accordance with clause 8 EN 1367-2
- 8) Prepare sample according to clause 7 of EN 933-8. Carry out test according to EN 933-8+A1, Appendix A.

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:

- a) preparing the material for the preparation of samples from one bearing,
- b) the distribution of bodies made of more bearings so as to ensure homogeneity of bodies in the field of testing of related characteristics,
- c) by review the material before releasing participants.

2.3 Instructions for the Elimination of Main Error Sources

PTP participants have the obligation:

- to handle the proficiency testing materials in the same way they handle the majority of routinely tested specimens,
- 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 label,
- to send the test results obtained during proficiency testing, including measurement uncertainties, to the PT Provider by the set deadline the participant received in the confirmation e-mail.

2.4 PTP Schedule

All other information, forms and records not included in this document are accessible in updated form at <http://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 [5], 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 [3], 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 $i = 1, \dots, p$, 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 C 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:

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

where n_i is the number of test results from the determination of one quantity in the i -th laboratory, y_k is the k -th value and \bar{y}_i 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

$$\bar{x} = \frac{1}{p} \sum_{i=1}^p x_i \quad (5)$$

is the arithmetic mean of the observed feature. The observed feature can be the average value of the quantity determined within the laboratory.

Furthermore,

$$s = \sqrt{\frac{1}{p-1} \sum_{i=1}^p (x_i - \bar{x})^2} \quad (6)$$

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:

$$G_1 = \frac{\bar{x} - x_1}{s}. \quad (7)$$

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:

$$h_i = \frac{\bar{y}_i - \bar{\bar{y}}}{\sqrt{\frac{1}{p-1} \sum_{i=1}^p (\bar{y}_i - \bar{\bar{y}})^2}}, \quad (8)$$

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 Interlaboratory Consistency Statistic - k

The interlaboratory consistency statistic k is calculated from the equation:

$$k_i = \frac{s_i \sqrt{p}}{\sqrt{\sum_{i=1}^p s_i^2}}, \quad (9)$$

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 Dispersion 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 Dispersion

$$s_r^2 = \frac{\sum_{i=1}^p (n_i - 1) s_i^2}{\sum_{i=1}^p (n_i - 1)} \quad (10)$$

3.3.2 Interlaboratory Dispersion

$$s_L^2 = \frac{s_d^2 - s_r^2}{\bar{n}}, \quad (11)$$

where

$$s_d^2 = \frac{1}{p-1} \sum_{i=1}^p n_i (\bar{y}_i - \bar{\bar{y}})^2 = \frac{1}{p-1} \left[\sum_{i=1}^p n_i (\bar{y}_i)^2 - (\bar{\bar{y}})^2 \sum_{i=1}^p n_i \right] \quad (12)$$

and

$$\bar{n} = \frac{1}{p-1} \left[\sum_{i=1}^p n_i - \frac{\sum_{i=1}^p n_i^2}{\sum_{i=1}^p n_i} \right]. \quad (13)$$

3.3.3 Reproducibility Dispersion

$$s_R^2 = s_r^2 + s_L^2, \quad (14)$$

where s_r^2 stands for repeatability dispersion and s_L^2 for interlaboratory dispersion.

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:

$$r = 2.8 s_r, \quad (15)$$

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:

$$R = 2.8 s_R, \quad (16)$$

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 [3] using the statistical methods described in ISO 13528 [8] and ISO 5725-5 [7]. The assigned value X is therefore determined as a robust estimate of the average value x^* (the A algorithm mentioned in [8] and [7]):

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

$$x^* = \text{the median of } x_i \text{ values } (i = 1, 2, \dots, p),$$

$$s^* = 1.483 \times \text{median of values } |x_i - x^*|.$$

The values of x^* and s^* are then processed as follows. First, $\varphi = 1.5 s^*$ is computed. For every x_i value ($i = 1, 2, \dots, p$), 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:

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

and

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

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:

$$u_x = 1.25 \times s^* / \sqrt{p}, \quad (20)$$

where s^* is a robust standard deviation determined by the above discussed algorithm.

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 $H = \frac{\text{int}\left(\frac{p+1}{2}\right)}{2}$ or $H = \frac{\text{int}\left(\frac{p+1}{2} + 1\right)}{2}$.

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 $t_{L,1-\alpha}(p)$ 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 [3] standard, 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:

$$z_i = \frac{|\bar{x}_i - x^*|}{s^*}, \quad (18)$$

where x^* is a robust estimate of the average value and s^* is a robust standard deviation determined according to 2.5.

the ζ-score (zeta-score) is calculated using the equation:

$$\zeta_i = \frac{|\bar{x}_i - x^*|}{\sqrt{u_i^2 + u_x^2}}, \quad (19)$$

where u_x is standard uncertainty of the assigned value determined according to 2.5. and 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 [31].

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

$ z \leq 2$	shows that the laboratory performance is satisfactory and generates no signal;
$2 < z \leq 3$	shows that the laboratory performance is questionable and generates an action signal;
$3 < z $	shows that the laboratory performance is unsatisfactory and generates an action signal.

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. References

6.1 Appendices

Appendix 1 Measurement Record

6.2 Related Internal Documents and Records

- [1] Quality Handbook of the PT Provider at the Institute of Building Testing, FCE BUT
- [2] Cancellation and Complaint Proceedings available at www.fce.vutbr.cz/szk

6.3 Standards

- [3] EN ISO/IEC 17043: Concordance Assessment – General Requirements for Proficiency Testing, ČNI 2010.
- [4] ISO 5725-1: Accuracy (Correctness and Concordance) of Methods and Measurement Results – Part 1: General Principles and Definitions, ČNI 1997.
- [5] ISO 5725-2: Accuracy (Correctness and Concordance) of Methods and Measurement Results – Part 1: A Basic Method for Determination of Repeatability and Reproducibility of a Normalized Measurement Method, ČNI 1997.
- [6] ISO 3534-1: Statistics. A Dictionary and Symbols – Part 1: Probability and General Statistical Terms, ČNI 1994.
- [7] ISO 5725-5: Accuracy (Correctness and Concordance) of Methods and Measurement Results – Part 5: Alternative Methods for Determination of Concordance of a Normalized Measurement Method, ČNI 1999.
- [8] ISO 13528 Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons, ISO 2005.
- [9] EN 933-1 Tests for geometrical properties of aggregates - Part 1: Determination of particle size distribution - Sieving method, 2012
- [10] EN 933-2 Tests for geometrical properties of aggregates - Part 2: Determination of particle size distribution - Test sieves, nominal size of apertures, 1997
- [11] EN 933-3 Tests for geometrical properties of aggregates - Part 3: Determination of particle shape - Flakiness index, 2012
- [12] EN 933-4 Tests for geometrical properties of aggregates - Part 4: Determination of particle shape - Shape index, 2008
- [13] EN 933-5 Tests for geometrical properties of aggregates - Part 5: Determination of percentage of crushed and broken surfaces in coarse aggregate particles, 1998
- [14] EN 933-8 Tests for geometrical properties of aggregates - Part 8: Assessment of fines - Sand equivalent test, 2012
- [15] EN 933-9 Tests for geometrical properties of aggregates - Part 9: Assessment of fines - Methylene blue test, 2013
- [16] EN 933-10 Tests for geometrical properties of aggregates - Part 10: Assessment of fines - Grading of filler aggregates (air jet sieving), 2010

- [17] EN 933-11 Tests for geometrical properties of aggregates - Part 11: Classification test for the constituents of coarse recycled aggregate, 2009
- [18] EN 1097-1 Tests for mechanical and physical properties of aggregates - Part 1: Determination of the resistance to wear (micro-Deval), 2011
- [19] EN 1097-2 Tests for mechanical and physical properties of aggregates - Part 2: Methods for the determination of resistance to fragmentation, 2010
- [20] EN 1097-3 Tests for mechanical and physical properties of aggregates - Part 3: Determination of loose bulk density and voids, 1999
- [21] EN 1097-5 Tests for mechanical and physical properties of aggregates - Part 5: Determination of the water content by drying in a ventilated oven, 1998
- [22] EN 1097-6, Tests for mechanical and physical properties of aggregates - Part 6: Determination of particle density and water absorption, 2001
- [23] EN 1097-7 Tests for mechanical and physical properties of aggregates - Part 7: Determination of the particle density of filler - Pyknometer method, 2008
- [24] EN 13450 Aggregates for railway ballast, 2004
- [25] EN 1367-1 Tests for thermal and weathering properties of aggregates - Part 1: Determination of resistance to freezing and thawing, 2007
- [26] EN 1367-2 Tests for thermal and weathering properties of aggregates - Part 2: Magnesium sulfate test, 2010
- [27] EN 1367-3 Tests for thermal and weathering properties of aggregates - Part 3: Boiling test for "Sonnenbrand basalt", 2001
- [28] EN 12620 Aggregates for concrete, 2008
- [29] ČSN 72 1179 Stanovení reaktivnosti kameniva s alkáliemi, ÚNMZ, 1968

6.4 Related External Documents

- [30] MPA 20 – 01 - . . for application of ČSN EN ISO/IEC 17043 Concordance Assessment – General Requirements for Proficiency Testing in the Accreditation System of the Czech Republic.
- [31] EA 4/02: Expressing of Measurement Uncertainties during Calibrations, 2000.EN 196-1 Cement Testing Methods - Part 1: Determination of Strength, ÚNMZ, 2005
- [32] TP MD 137, Vyloučení alkalické reakce kameniva v betonu na stavbách PK (+ změna 1), MD-OPK č.j. 509/03-120-RS/1 / 1. november 2003 revision 2011, available from http://www.pjpk.cz/TP%20137_1.pdf