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Differences in the accuracy of test methods in the field of testing aggregate and determination of their causes

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Abstract. The article is based on the authors’ many years of experience in providing laboratory proficiency testing including organization of proficiency testing by interlaboratory comparison. In the introduction are compared the basic tests in the field of aggregates, based on their coefficient of variation. Another part details the influence of each testing stage (sampling, sample preparation and the test itself) on the accuracy of the test results and proposes possible measures to reduce their variance.

1. Introduction
The main goal of laboratory testing is to determine with adequate accuracy the desired properties of materials and structures. This accuracy then decides subsequent designs, calculations, evaluations, or result interpretations, which affect the general outcome of a construction or its part. This is why it is necessary to limit or eliminate all parts of a testing procedure that could have a negative impact on the accuracy of the results.

The proportioning of the stages of a particular testing procedure can be ascertained from data obtained in proficiency testing programmes aimed at testing aggregates and organised by ASPK, s.r.o. Brno and by the Proficiency Testing Provider at SZK FAST, Brno University of Technology between 1995 and 2017. Their statistical evaluation is performed in accordance with CSN ISO 5725 “Accuracy (trueness and precision) of measurement methods and results” [11].

The paper uses the following definitions:
- **Accuracy**: the closeness of agreement between a test result and an accepted reference value.
- **Trueness**: the closeness of agreement between the mean value of a great number of test results and an accepted reference value μ.
- **Bias**: the difference between the mean value of test results m and an accepted reference value μ.
- **Precision**: the closeness of agreement between independent test results under predefined conditions (reproducibility).
- **Reproducibility**: precision under reproducibility conditions.
- **Reproducibility conditions**: conditions under which test results are arrived at by the same method, using the same test specimens, in different laboratories, and by different testers using different equipment.

These definitions allow the acceptably simplified statement that the quantity which determines the accuracy of a test method is its reproducibility. It is, however, not possible to compare the values of reproducibility without taking the reference value into account.

A comprehensive evaluation was thus performed using the reproducibility coefficient of variation defined by CSN ISO 3534-1 "Statistics - Vocabulary and symbols - Part 1: General statistical terms and terms used in..."
probability” [13] as a ratio of reproducibility to the mean value of the quantity being measured. Figure 1 below shows the values of variation coefficient for selected aggregate tests measured as part of proficiency testing programmes from 1996 to 2017.

![Figure 1. Determination of the variation coefficient in selected tests of aggregate.](image)

The following section describes the influence that each stage of testing (sampling, specimen preparation, and the testing itself) has on the accuracy of test results.

2. Division of testing activities

Laboratory testing encompasses all activities connected with carrying out tests including sampling and specimen preparation.

2.1. Sampling

Sampling is a standard procedure, during which a part of the material or product is taken so that it provides a representative sample for the purposes of testing CSN EN 932-1 [4].

2.2. Sample preparation and adjustment

Sample preparation is a procedure during which the representative sample is adjusted (homogenized and reduced) so as to achieve a test (laboratory) sample, see CSN EN 932-2 [5].

| Activity            | Standard                                                                 | Percentage of testing laboratories to perform said activity |
|---------------------|--------------------------------------------------------------------------|------------------------------------------------------------|
| Sampling            | Tests for general properties of aggregates Part 1: Methods for sampling CSN EN 932-1 | 75 %                                                       |
| Sample preparation  | Tests for general properties of aggregates Part 2: Methods for reducing laboratory samples CSN EN 932-2 | 10 %                                                       |

2.3. Testing

Testing is a procedure during which the test sample is tested for the required or observed properties. This activity has been extensively explored and documented, which is why this paper does not address it in detail.

3. Determination of the influence of individual operations on the overall test result

According to CSN ISO 11648-1 [12] each process has its own variance component:

- the variance component of sampling caused by the taking of partial samples,
- the variance component of sample preparation produced during adjusting and reducing the sample,
• the variance component of measurement characterising the precision of the method used.

The percentage of each component for the overall result of a test method is quantitatively determined using differences between the proficiency testing programmes from each of the above-listed providers. The differences lie in the way of sample preparation, making it possible to estimate the percentage of each component in the overall result. The specification of the influence of sample preparation by homogenization and reduction on the determination of particle size distribution was supplemented by the authors’ own measurement in cooperation with laboratories at COLAS CZ, a.s. and M.I.S. a.s. The results are discussed in section 4.

3.1. Determination of particle size distribution - sieving method

The testing of particle size distribution according to CSN EN 933-1 [6] underwent a change in 2005 in the way samples are prepared within proficiency testing programmes, where the test sample is centrally prepared from narrow fractions of aggregate and supplied to the laboratories at an amount equal to a standard testing batch (2.63 kg for fraction 0/16). Thanks to this, the variance components of a) sampling and b) sample preparation are eliminated. Figure 2 shows the difference.

![Figure 2](image)

Figure 2. Determination of reproducibility for the test according to CSN EN 933-1 using a 4 mm sieve.

3.2. Determination of particle shape - shape index

The determination of particle shape - shape index according to CSN EN 933-4[7] entailed comparing the results of standard IC with IC from 2008 and 2017, which used identical samples - the variance components a) and b) were thus eliminated. Figure 3 shows the difference.

![Figure 3](image)

Figure 3. Determination of reproducibility for the test according to CSN EN 933-4 using fraction 8/16

Note: Proficiency testing programmes implemented thus far still lack sufficient knowledge and documentation for a possible determining of the influence of the components of the testing process for tests according to CSN EN 933-8 [8], CSN EN 933-9 [9], CSN EN 1097-2 [10] and CSN EN 1097-6 [11].
4. Determination of the influence of sample preparation by homogenization and reduction for determining particle size distribution

The next step for eliminating the influence of variance components is ascertaining the percentage of influence that the variance component of sampling and variance component of sample preparation have. This step was carried out only for the determination of particle size distribution CSN EN 933-1[6]. Two laboratory samples (fraction 0/16, mass 2.640 kg) were mixed to form a single sample (fraction 0/16, mass 5.280 kg). A laboratory sample was then taken from it by two ways:

- reduction and homogenization according to CSN EN 932-2 [5],
- by simply taking a sample.

4.1. Homogenized samples

**Table 2.** Sieve analysis of the homogenized laboratory sample, fraction 0/16.

| Lab. no./sieve | 16 | 8  | 4  | 2  | 1  | 0.50 | 0.25 | 0.125 | 0.063 |
|----------------|----|----|----|----|----|------|------|-------|-------|
| A/1            | 100| 59 | 30 | 20 | 12 | 8    | 5    | 4     | 1     |
| A/2            | 100| 59 | 30 | 19 | 12 | 8    | 5    | 3     | 1.9   |
| A/3            | 100| 58 | 31 | 20 | 12 | 9    | 5    | 3     | 1.9   |
| A/4            | 99 | 59 | 30 | 19 | 11 | 8    | 4    | 3     | 1.6   |
| m              | 99.8| 58.8| 30.5| 19.5| 11.8| 8.3  | 4.5  | 2.8   | 1.7   |
| s. d.          | 0.50| 0.50| 0.58| 0.58| 0.50| 0.50 | 0.58 | 0.50  | 0.24  |
| B/5            | 99 | 59 | 30 | 19 | 13 | 8    | 4    | 3     | 1.5   |
| B/6            | 99 | 59 | 30 | 20 | 12 | 8    | 4    | 3     | 1.4   |
| B/7            | 99 | 59 | 30 | 12 | 12 | 8    | 4    | 3     | 1.7   |
| B/8            | 99 | 59 | 30 | 20 | 12 | 8    | 4    | 3     | 1.5   |
| m              | 99.0| 58.8| 30.8| 19.8| 12.3| 8.0  | 4.0  | 3.0   | 1.5   |
| s. d.          | 0.00| 0.50| 0.50| 0.50| 0.50| 0.00 | 0.00 | 0.00  | 0.13  |
| C/9            | 100| 57 | 31 | 19 | 12 | 9    | 5    | 4     | 2.1   |
| C/10           | 100| 58 | 31 | 19 | 12 | 8    | 5    | 3     | 1.9   |
| C/11           | 100| 59 | 31 | 12 | 12 | 8    | 5    | 3     | 1.9   |
| C/12           | 100| 59 | 31 | 20 | 12 | 8    | 5    | 3     | 1.9   |
| m              | 100.0| 58.5| 30.8| 19.5| 12.0| 8.3  | 5.0  | 3.3   | 2.0   |
| s. d.          | 0.00| 0.58| 0.50| 0.58| 0.00| 0.50 | 0.00 | 0.50  | 0.10  |

Legend  m general mean value  s.d. standard deviation

4.2. Non-homogenized samples

**Table 3.** Sieve analysis of a non-homogenized sample, fraction 0/16.

| Lab. no./sieve | 16 | 8  | 4  | 2  | 1  | 0.50 | 0.25 | 0.125 | 0.063 |
|----------------|----|----|----|----|----|------|------|-------|-------|
| A/1            | 97 | 57 | 31 | 19 | 12 | 8    | 5    | 4     | 1.3   |
| A/2            | 98 | 58 | 28 | 20 | 13 | 8    | 5    | 3     | 1.5   |
| A/3            | 100| 57 | 30 | 18 | 12 | 7    | 3    | 2     | 0.6   |
| A/4            | 97 | 56 | 29 | 19 | 12 | 8    | 5    | 3     | 1.5   |
| m              | 98.0| 57.0| 29.5| 19.0| 12.3| 7.8  | 4.5  | 3.0   | 1.2   |
| s. d.          | 1.41| 0.82| 1.29| 0.82| 0.50| 0.50 | 1.00 | 0.82  | 0.43  |
| B/5            | 100| 57 | 30 | 19 | 12 | 8    | 5    | 3     | 1.6   |
| B/6            | 97 | 57 | 29 | 19 | 11 | 8    | 5    | 3     | 1.8   |
| B/7            | 99 | 58 | 31 | 20 | 13 | 7    | 5    | 2     | 1.4   |
| B/8            | 98 | 58 | 30 | 19 | 12 | 8    | 5    | 3     | 0.5   |
| m              | 98.5| 57.5| 30.0| 19.3| 12.0| 7.8  | 5.0  | 2.8   | 1.3   |
| s. d.          | 1.29| 0.58| 0.82| 0.50| 0.82| 0.50 | 0.00 | 0.50  | 0.57  |
| Lab. no./sieve | 16 | 8  | 4  | 2  | 1  | 0.50 | 0.25 | 0.125 | 0.063 |
5. Result discussion

Available literature [16-18] does not address this question. We believe that this is largely due to the fact that the test sample batch was approximately twice the size; in the case of fraction 0/16, it was at least 5 kg, see ČSN 72 1172 [15] compared to 2.6 kg according to ČSN EN 933-1 [6]. The number of measurements is also different: ČSN 72 1172 [15] requires two, ČSN EN 933-1[6] considers one measurement to be enough.

Until quite recently, one test of aggregate particle size distribution (fraction 0/16) thus required a minimum of 10 kg, today it is only 2.6 kg; i.e. about one quarter. Such a significant reduction in testing sample size causes both preparatory stages (sampling and later homogenisation) to have a much more dramatic effect on the test result. Data shown in graphs G2, G3, and G4 can be used to partially determine the percentage of each variance component in the overall result; at the moment, however, only the influence of the variance component of the test itself and the compound effect of the variance component of sampling and sample preparation.

The results are shown in table 4.

| Test method | R normal | R reduced | SR a+b+c % | SR c % | SR a+b % |
|-------------|----------|-----------|------------|--------|----------|
| Particle size distribution ČSN EN 933-1 | 4.5 | 2.1 | 100 | 46 | 54 |
| Determination of the shape index SI ČSN EN 933-4 | 9.1 | 3.6 | 100 | 39 | 61 |

Legend
R normal reproducibility during normal testing %
R reduced reproducibility with elimination of sampling and laboratory sample preparation %
SR a+b+c influence of variance components a+b+c (total) = 100%
SR c influence of variance components c (test itself)
SR a+b influence of variance components a+b (sampling and sample preparation)

Data shown in tables 2 and 3 can be used to determine the percentage of the influence of sample homogenization on the overall result. The result is in table 5.

| Sieve (mm) | 8 | 4 | 2 | 1 | 0.5 | 0.25 | 0.125 | 0.063 | average |
|------------|---|---|---|---|-----|------|-------|-------|--------|
| RH | 1.59 | 1.48 | 1.65 | 1.20 | 1.17 | 1.60 | 1.39 | 0.71 | 1.35 |
| RN | 4.15 | 4.98 | 2.64 | 2.65 | 1.56 | 1.90 | 1.85 | 1.43 | 2.65 |
| RH/RN % | 38 | 30 | 63 | 45 | 75 | 84 | 75 | 50 | 51 |

Legend
RH reproducibility of the homogenized sample %
RN reproducibility of the non-homogenized sample %

The average reproducibility of tests performed on the non-homogenized sample is 2.65 %, while tests with the homogenized/reduced sample returned a value of 1.35 %.

Using this data helps to clarify the influence (variance components) of each operation on the overall test result. The results are listed in table 6.
Table 6. Determination of the influence of each operation on the test result according to EN 933-1[6].

| Process                              | methodology            | influence % |
|--------------------------------------|------------------------|-------------|
| Sampling                             | CSN EN 932-1           | 27          |
| Laboratory sample preparation        | CSN EN 932-2           | 27          |
| Testing                              | CSN EN 933-1           | 46          |

6. Conclusion

The above-presented data shows that the influence of sampling and subsequent preparation (homogenisation) of a test sample on the test result is considerable and is often neglected in contemporary laboratory testing. The actual testing receives far more attention, which, however, takes only a 50% part in the accuracy of the result. It is probable that in other tests, or areas of testing, this ratio could be different, but in no case is it strongly significant (100%). It must be noted that making an effort to improve this (i.e. reducing the variance components of reproducibility for the whole process) carries no extra material costs and requires no time-consuming and complicated testing procedures. All that is necessary is to thoroughly follow existing standards. As concerns meeting the requirements of [18] and [19]; i.e. standards on Factory Production Control, the scope of further required tests may be limited. Consistent application and compliance with the proposed model will improve the accuracy of test results. This will allow for more precise design of components (such as road layers) or entire structures. With the annual cost of construction in the order of CZK 100 billion, improving the precision of a testing/monitoring activities, resulting savings of 1% can be considered very significant.

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