EA Expert Group on Uncertainty of Measurement in Testing
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EXAMPLE FOR ESTIMATING THE MEASUREMENT UNCERTAINTY IN BUILDING MATERIALS TESTING (AGGREGATES)

Background

The first draft of the 'Guide to the Expression of Uncertainty in Measurement' (GUM) was published in January 1993. Together with the United Kingdom Accreditation Service (UKAS, the former NAMAS) the author of the following example has used the general rules of the GUM in 1993/4 to apply them to testing purposes. The examples produced in that project have been published [1]. The methodical approach used has generally been described by Morkowski as the 'controlled assessment of the uncertainty of the result' [2]. This procedure, which is one out of five, has also been applied for the following calculation. It has been discussed in the EA Expert Group on Uncertainty of Measurement in Testing. On its last meeting in January 2003 the group decided to publish the document for debate.

Introduction

Concrete and road construction handle aggregates in large quantities. Their properties are determined in geometrical, mechanical and chemical tests. One of the criteria for their use is the shape of the fragments. Particles of a more cubical shape are in many cases easier and more cost-effective to process than those with a flat or longish form. Moreover, in concrete construction the need for binders increases when using particles with larger surfaces which in turn involve higher materials costs.

A test method on how to quantitatively determine the proportion of unfavourably shaped particles is described in EN 933-3. The materials are first sieved on metal plates with square holes and then on bar sieves. Particles with a clearly smaller height in comparison to length and/or width, i.e. flat or longish aggregates, pass the bar sieve. Their percentage by mass of the total constitutes the Flakiness index FI.

The model of the measurement process is given by the formula

$$FI = \frac{Mass_{observed}}{Mass_{total}} \cdot 100 \ \pm \text{corrections}$$

In the annex is an example of a test result. The material of the size fraction 8/16 mm has an overall Flakiness index of FI = 9 M.-%, for which the uncertainty is unknown and shall be determined in the following. For determining the uncertainty or the correction according to the model the test process has been subdivided into nine single steps:

No. of the single step	Step of test procedure
1	Sampling and reduction of samples
2	Drying of test sample
3	Weighing of test sample
4	Sieving on square holes in defined size fractions
5	Weighing of single size fractions
6	Sieving of each size fraction on bar sieves
7	Weighing of passing masses of each size fraction
8	Calculation of results for each size fraction
9	Statement of results

Step 1 (Sampling and reduction of samples)

Data on uncertainty of sampling and reduction of samples were not available. Therefore four trained persons took a multiple of the required material from a typical pile and reduced it to the required test sample quantity. The uniformity of sampling and reduction of samples was determined on a bar sieve by comparing the two sample quantities last divided. The following passing masses in M.-% were determined:

Sample No.	Sample A_i	Sample B_i	Mean value M_i
1	37,92	38,50	38,21
2	37,21	38,31	37,76
3	36,40	39,39	37,90
4	38,52	36,94	37,73

To calculate the estimated standard deviation associated with this input value the average standard deviation is used ($u(x_i)$ denoted as u_i):

$$u_1 = \sqrt{\frac{1}{n-1} \cdot \sum_{i=1}^{n} \left[(A_i - M_i)^2 + (B_i - M_i)^2 \right]}$$

$$u_1 \approx 1{,}5 \text{ M.-\%}$$

Step 2 (Drying of the test sample)

During testing and storage in the laboratory aggregates may absorb water subject to temperature and humidity and may thus change their mass. According to the test specification the sample has to be dried at a raised temperature, until it reached mass stability. The sample is considered to be dry, if for two consecutive weighings at intervals of one hour the masses do not differ by more than 0,1 %.

Provided that the test is executed within a short period of time the environmental conditions practically do not affect the mass of the sample. The uncertainty associated with this factor is negligible:

$$u_2 \leq 0{,}1 \text{ M.-\%}$$

Step 3 (Weighing of the test sample)

The weighings performed in this step of test procedure do not have a direct influence on the test result. As the material loss is very small (annex) a further consideration of a partial uncertainty u_3 is not required:

$$u_3 \leq 0{,}1 \text{ M.-\%}$$

Step 4 (Sieving on square holes in defined size fractions)

In sieving one must distinguish between a separation in defined size fractions (partial step 4A) and the influence of particles of critical size (partial step 4B).

Partial step 4A (Separation in size fractions):

In the test the three size fractions 12,5/16 mm, 10/12,5 mm and 8/10 mm are quantified as well as oversized particles (> 16 mm) and undersized particles (< 8 mm). As the overall Flakiness index gives a sum result it is of no importance which size fraction contains which particle shape portion. An uncertainty contribution of this partial step 4A can therefore be neglected:

$$u_{4A} \leq 0{,}1 \text{ M.-\%}$$

Partial step 4B (Particles of critical size):

Particles of critical size are single aggregates which either just pass the respective sieve or which are a little too large for the sieve openings and therefore remain as a residue. Particles of critical size arise from the tolerance of the sieve openings and from differences in the performance of the test, such as the duration of sieving and the way of moving the sieves.

Standard test sieves shall comply with the tolerance ranges of (8 ± 0,19) mm or (16 ± 0,27) mm. A particle with a dimension of about 16,27 mm could just pass the 16mm sieve – although it theoretically represents an oversized particle – and thus affect the overall result. Something similar applies to a particle with a dimension of 8,19 mm, which could be classified as an undersized particle. The more comparable the opening widths of the used sieves are, the smaller is the probability of different sieving results. To quantify this statement the following assumption is made:

The mass of the residue remaining on the 16mm sieve was 56,1 g (see annex). This residue could theoretically be composed of 6 cubic particles with an average mass of m_S = 9,35 g and edge lengths of 15,96 mm. (A density of ρ = 2,300 g/cm^3 is assumed.) It would then exclusively consist of particles of critical size. However, such an assumption would be inconsistent with testing experiences. Consequently the oversized particles from the test were measured separately. Two of five particles had dimensions within a range of (16 ± 0,27) mm and were therefore considered as particles of critical size. As such particles could partly be contained in the fraction of 12,5/16 mm, an assumption of four particles of critical size is realistic. Four particles with an edge length of 16 mm have an overall mass of 37,7 g. In this case the size fraction $d_{12,5}/D_{16}$ (247,0 g, annex) will show extreme values between 228,2 g and 265,8 g (range 2a = 37,7 g). It is most unlikely that a sieve has such a uniform pattern of openings. In the majority of cases openings around the nominal opening width are much more probable. Hence a triangular distribution (b = 6) is chosen.

$$u_{4B} = \frac{a}{\sqrt{b}} \cdot \frac{1}{M_1}$$

$$u_{4B} \approx 0{,}8 \text{ M.-\%}$$

These considerations concern coarse aggregates. Influences resulting from the sieving process can be neglected. The 8mm sieve is no longer considered, as the masses are much smaller in comparison to those on the 16mm sieve.

Step 5 (Weighing of single size fractions)

The test specification requires a balance with an error margin of ± 0,1 % related to M_0. The laboratory ensured that the balance used met these requirements with ± 0,05 % of M_0 and k = 3. That means for the uncertainty associated with the factor u_5' is:

$$u_{5-i} = \frac{M_0 \cdot 0{,}0005}{k} \cdot \frac{1}{R_i}$$

$$u_{5-1} = 0{,}0728 \text{ M.-\%}$$
$$u_{5-2} = 0{,}0542 \text{ M.-\%}$$
$$u_{5-3} = 0{,}0409 \text{ M.-\%}$$

$$u_5' = \sqrt{u_{5-1}^2 + u_{5-2}^2 + u_{5-3}^2}$$

$$u_5' \approx 0{,}1 \text{ M.-\%}$$

The weighing results correlate because always the same balance was used. In spite of a small additional contribution due to the correlation u_5' ($u_5 \approx 0{,}17$ M.-%) this standard uncertainty can be neglected.

Step 6 (Sieving of each size fraction on bar sieves)

According to the test standard the widths of the sieve slots may vary by ± 0,1 mm. For the size fraction of 12,5/16 mm a bar sieve with 8 mm slot width is used. The assumption of an oversized particle (cuboid shape with a length and width of 16 mm as well as a height of 8 mm and a density of 2,300 g/cm^3) results in a mass of 4,7 g (range 2a = 4,7 g). As the total number of residue particles is small the number of particles of critical size must be even smaller. Hence it is assumed that only in half of all tests a particle of critical size occurs (p = 0,5). Furthermore in this case a rectangular distribution (b = 3) is more plausible than a triangular one because it only concerns single aggregates.

$$u_6 = \frac{(p \cdot a)}{\sqrt{b}} \cdot \frac{1}{M_2}$$

$$u_6 \approx 0{,}8 \text{ M.-\%}$$

A noticeably smaller standard uncertainty would arise for undersized particles which shall no further be considered. Again the influence of the ‚intermediate test results' can be neglected (see partial step 4A).

Step 7 (Weighing of the passing masses of each size fraction)

In this step three weighings are performed on the masses passing the bar sieves. The calculation is made analogously to step 5:

$$u_{7-i} = \frac{M_0 \cdot 0{,}0005}{k} \cdot \frac{1}{m_i}$$

$$u_{7-1} = 0{,}687 \text{ M.-\%}$$
$$u_{7-2} = 0{,}618 \text{ M.-\%}$$
$$u_{7-3} = 0{,}490 \text{ M.-\%}$$

$$u_7' \approx 1{,}1 \text{ M.-\%}$$

Step 8 (Calculation of the results for each size fraction)

The results for the individual size fractions are summarized in the table in the annex as masses R_i. The records are made with two decimals which is quite precise. The associated standard uncertainty u_8 can therefore be neglected:

$$u_8 \le 0{,}1 \text{ M.-\%}$$

Step 9 (Statement of result)

According to the test specification the overall Flakiness index has to be rounded to the next integer. Consequently the range is 2a = 1. The probability is the same for results between –0,5 and +0,5, so that a rectangular distribution (b=3) is assumed:

$$u_9 = \frac{a}{\sqrt{b}}$$

$$u_9 \approx 0{,}3 \text{ M.-\%}$$

The standard uncertainty u_9 can be neglected as it is much smaller than other contributions to the overall uncertainty.

Calculation of the combined standard uncertainty

Only some of the components of the combined measurement uncertainty are of importance for further calculations:

No.	Step of test procedure	Standard uncertainty
1	Sampling and reduction of samples	$u_1 \approx 1{,}5$ M.-%
4	Sieving on metal plates with square holes	$u_{4B} \approx 0{,}8$ M.-%
6	Sieving on bar sieves	$u_6 \approx 0{,}8$ M.-%
7	Weighings of passing masses through bar sieves	$u_7 \approx 1{,}1$ M.-%*

*with correlation of single components

For calculating the combined measurement uncertainty from these data the correlated quantities have to be considered (equation 13 of GUM):

$$u_c(y) = \sqrt{\sum_{i=1}^{N}\left[\frac{\partial f}{\partial x_i}\right]^2 u^2(x_i) + 2\sum_{i=1}^{N-1}\sum_{j=i+1}^{N}\frac{\partial f}{\partial x_i}\frac{\partial f}{\partial x_j}u(x_i, x_j)}$$

All these considerations are made on an approximation basis as no complete model is available. Data for quantification of $\left[\frac{\partial f}{\partial x_i}\right] = c_i$ and $\left[\frac{\partial f}{\partial x_j}\right] = c_j$ are missing analogously. As far as no inconsistencies arise from the following plausibility considerations, it is assumed that c_i and c_j equal 1.

For all weighings the same weighing instrument was used. For this reason the correlation coefficient can be assumed to be +1. Hence the calculation formula is shortened to

$$u_c = \sqrt{\sum_{i=1}^{N} u^2(x_i) + 2\sum_{i=1}^{N-1}\sum_{j=i+1}^{N} u(x_i)\cdot u(x_j)}$$

or

$$u_c = \sqrt{u_1{}^2 + u_{4B}{}^2 + u_6{}^2 + u_7'^2 + 2\cdot\left[(u_{7-1}\cdot u_{7-2}) + (u_{7-1}\cdot u_{7-3}) + (u_{7-2}\cdot u_{7-3})\right]}$$

$$u_c = 2{,}55 \text{ M.-\%}$$

Expanded uncertainty

$$U = k \cdot u_c$$

$$\boxed{U = 5{,}1 \text{ M.-\%}}$$

This expanded uncertainty is stated as the standard measurement uncertainty multiplied by the coverage factor k = 2. The distribution function is unknown, but if the distribution is normal (Gaussian distribution) k = 2 corresponds to a coverage probability of approximately 95 %.

Precision data

In its informative annex B EN 933-3 provides data for both the repeatability limit r and the reproducibility limit R. For values of FI between 8 and 20 the given values are r = 2,8 and R = 5.

The figures of the reproducibility limit and the calculated expanded measurement uncertainty are in a comparable range. The experimental data which are based on procedures according with ISO 5725 thus confirm the underlying model - in spite of the very different mathematical approaches. However there is an important difference:

The presumptions for the testing conditions for the above calculation are valid for a specific laboratory situation. The expanded uncertainty should therefore be expected to lie between r and R. Additionally one must consider that the precision data given do not fully encompass both sampling and reduction. It can therefore be said that the above calculated expanded measurement uncertainty represents a plausible figure.

The value of the calculation would be very limited if it were only used for the confirmation of the underlying model. GUM-based calculations of measurement uncertainty can be used more specifically than the general figures repeatability limit r and reproducibility limit R. This example both quantifies the contribution of sampling and reduction on the overall measurement uncertainty and specifies the crucial steps in procedure as regards uncertainty. These data are necessary when the uncertainty of test results shall be optimized and they are essential when the test procedure undergoes changes.

Literature

1) Hinrichs, W.
   Uncertainty of Measurement; Example: Construction Materials Testing
   Materialprüfung 36 (1994) 11/12, 476-480

2) Morkowski, J.S.
   Characterisation and validation of test methods
   Proceedings of the 3rd Eurolab Symposium 446-462, Berlin 1996

Mass of test sample M_0 = 1079,4 g		Mass of residue remaining on 16-mm sieve = 56,1 g Mass of passing through 8-mm sieve = 3,2 g Sum of these masses = 59,3 g		
Sieving on square holes		**Sieving on bar sieves**		
Particle size d_i/D_i mm	Mass (R_i) of particle size d_i/D_i g	Nominal slot width of bar sieve mm	Mass passing through bar sieve (m_i) g	$FI_i = (m_i/R_i)x100$ M.-%
12,5 / 16	247,0	8	26,2	10,6
10 / 12,5	332,1	6,3	29,1	8,8
8 / 10	439,8	5	36,7	8,3
$M_1 = \Sigma R_i =$	1.018,9	$M_2 = \Sigma m_i =$	92,0	-
$FI = (M_2/M_1) \times 100 = 9$				
$100 \times \frac{M_0 - [\sum R_i + \sum (masses)]}{M_0} = 0,1\ \%$				