The evaluation of the measurement uncertainty of the thousand-seed weight in accredited testing laboratories

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Abstract
The evaluation of the measurement uncertainty of the seed quality indicators is an integral part of testing in accredited testing laboratories. The thousand-seed weight is one of the essential indicators, the limit values of which are not standardized, but are taken into account when determining the cost of a lot of seeds or seeding rates.

The developed method for evaluating the measurement uncertainty of the thousand-seed weight by measuring two weights of 500 seeds is presented in the paper. According to the analysis of a basic calculation formula, the impact of correctness and accuracy on the measurement uncertainty was assessed. It was shown that the major measurement uncertainty components are uncertainties of weighting and repeatability. The repeatability is advisable to be evaluated by Type B using the limit of repeatability, if the national standard specifies the testing method. The authors have analysed the conditions when other components can be neglected. The uncertainty budget and an example of its calculation were received.

Keywords: seed quality indicators; thousand-seed weight; seed certification; measurement uncertainty; accredited seed testing laboratory.

Received: 21.12.2022     Edited: 23.03.2023     Approved for publication: 29.03.2023

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The evaluation of the measurement uncertainty is an important part of the certification process and the overall quality assurance process in the food chain. The accuracy of measurements depends on many factors, which are ultimately defined as measurement uncertainty components.

The impact of measuring equipment is significant, however proper metrological assurance can minimize this effect. Measures to minimize this impact include, among other things, the introduction of an integrated hardware and software system designed for using in the continuous monitoring and recording of the seed laboratory operations [5].

The international seed health testing methods are published in International Rules for Seed Testing (ISTA Rules 2023) and are subject to changes to ensure their flexibility and efficiency. Ukrainian testing methods have remained unchanged since 2002 [4], which allows no changes to the methodological component of the combined measurement uncertainty.

The issue of the evaluation of measurement uncertainty during the sunflower seed testing, which is based on the “moisture mass fraction” indicator, was considered in [6]. Paper [7] presents the evaluation of measurement uncertainty when studying the quality and safety indicators of sunflower oil on the example of the oil content present in sunflowers seeds. The extraction method for the “oil content” indicator was used.

Features of estimating the thousand-seed weight indicators according to the International and Brazilian standards for forest species using image analysis are given in [8]. The use of an automatic seed testing system has increased the efficiency of testing and reduced human intervention. The study is dedicated to determining the reduction of testing time, but the issue of accuracy, including measurement uncertainty, is not considered.

Therefore, there is reason to believe that the difference between International Rules for Seed Testing and Ukrainian ones, and the lack of regulated methods for the evaluation of measurement uncertainty of the seed quality indicators make it important to conduct research in this area.

The aim of the study is to develop a method for the evaluation of measurement uncertainty of the quality indicator “thousand-seed weight”. This method, on one hand, should take into account all potential components of measurement uncertainty, and on the other hand, it should be as clear and convenient as possible for the use by the personnel of the seed testing laboratory.

3. Research materials and methods

The research was conducted at a seed laboratory that is accredited to meet the requirements of the standard [2]. The method of testing the thousand-crop seed weight is accredited according to the measurement methods, which are given in [4]. The method was tested for a sample of corn seeds of DKS CH (F1) cultivar produced in 2022.

The quality indicator “thousand-seed weight” is a mass of 1000 seeds, determined according to a standardized method, and expressed in grams. For testing, a sample of seeds of the main crop is used after analysing by the “purity” indicator. The standard [4] allows for manual and automatic keeping of records using seed counters.

Testing is performed using one of two methods: 1) eight replicates of 100 seeds; 2) two replicates of 500 seeds. This paper presents a method for the evaluation of the expanded uncertainty for the second case. According to the method, two replicates of 500 seeds are counted from the seeds of the main crop without selection, and each is weighed with the required accuracy. The missing number of seeds is taken from the second replicate during the purity analysis or from the average sample. The arithmetic mean of the masses of both replicates, their sum, and the actual difference between them are calculated. According to the requirements [4], the actual difference between replicates should not exceed 3% of the arithmetic mean.

The analysis is considered validated if the actual difference between replicates does not exceed 3% of the arithmetic mean. If the actual difference exceeds the permissible limit, a third test shall be performed. The ultimate result is calculated based on those two replicates, the actual difference between which is within acceptable limits.

If the value of all replicates exceeds the limits of permissible deviations, the arithmetic mean is calculated from all replicates, but in the absence of errors. Rounding is done to the first decimal place. For small-seeded crops, the thousand-seed weight of which is less than 10 g, rounding up is done to the second decimal place.

The estimation was carried out in accordance with the methods of the evaluation of expanded measurement uncertainty regulated by [3].

4. A method for the evaluation of the measurement uncertainty of the thousand-seed weight

The evaluation of the measurement uncertainty begins with an analysis of a basic calculation formula of the method.

The thousand-seed weight is determined using the readings of the balance.

\[ M = m_1 + m_2, \]  

where \( m_1 \) and \( m_2 \) are the weights of two samples of 500 seeds each, g.

The result model equation for calculating the measurement uncertainty is as follows:

\[ M_s = M + \delta m_1 + \delta m_2 + \delta_1 + \delta_2 + \delta_4, \]  

where \( \delta m_1 \) and \( \delta m_2 \) are the uncertainties of the weights of the samples, and \( \delta_1, \delta_2, \delta_4 \) are the uncertainties of the balance readings.

The estimation was carried out in accordance with the methods of the evaluation of expanded measurement uncertainty regulated by [3].

The thousand-seed weight is determined using the readings of the balance.

\[ M = m_1 + m_2, \]  

where \( m_1 \) and \( m_2 \) are the weights of two samples of 500 seeds each, g.

The result model equation for calculating the measurement uncertainty is as follows:

\[ M_s = M + \delta m_1 + \delta m_2 + \delta_1 + \delta_2 + \delta_4, \]
where $\delta_{m1}$ and $\delta_{m2}$ are the components of weighing measurement uncertainty, g; $\delta_c$ is a random component of the result, g; $\delta_p$ is a component of the correctness of the method, g; $\delta_d$ is a component of the rounding of the result, g.

The components of the measurement uncertainty for (1) are shown in the cause-effect diagram (Fig. 1).

4.1. Analysing the components of measurement uncertainty

Weighing measurement uncertainty. As it can be seen, the main source of the instrumental component of the measurement uncertainty in formula (2) is the balance. Weighting standard uncertainty is evaluated according to the internal regulatory document of a testing laboratory. Such a document can be work instructions, procedures, etc. Since both weights are weighed on the same balance, the input values are $\delta_{m1} = \delta_{m2}$. Given this, we denote the standard weighing measurement uncertainty as $u_{m}$, ($u_{m1} = u_{m2} = u_{m}$).

Random components of the measurement uncertainty $\delta_c$. The measurement uncertainty of counting has a discrete distribution of results. We assume that it is a component of the repeatability regulated by the standard [4]. The component arising from the heterogeneity is minimized during the sample preparation by homogenizing the sample before testing. We take into account that only Agronomist-Inspector (certification auditor) authorized by the Ministry of Agrarian Policy and Food of Ukraine are involved in the sampling process. Such auditors are supervised by both the Seed Certification Body and the Ministry of Agrarian Policy and Food. Therefore, we consider this component insignificant and do not take it into account in the combined measurement uncertainty. The component caused by previous tests (determining the “purity” indicator) is minimized by constant monitoring by the competent personnel in accordance with the requirements [2]. Therefore, we consider this component insignificant and do not take it into account in the combined standard measurement uncertainty.

The random component arises from the effect of counting 500 seeds, seed weight heterogeneity, sample heterogeneity and so on. Since testing is carried out only under regulated environmental conditions, we consider its influence on the measurement uncertainty of the test result to be taken into account in the value of the repeatability limit $r$. The mean of the random component $\xi$ is zero, but its contribution to the measurement uncertainty of the result is taken as the repeatability of the results $u_r$. Thus, we estimate the repeatability by Type B, since Type A evaluation of two results is invalid. It is known [9], that if the number of measurement results is less than 10, it is recommended to calculate the uncertainty of repeatability using the formula $s_1 = s \sqrt{\frac{n-1}{n-3}}$, where $s$ is the estimation of the standard deviation. However, given the number of repeated tests is $n = 2$, the formula cannot be applied in practice. The authors consider it appropriate to apply another estimation. The value of the measurement uncertainty, which is caused due to random factors, is the value of the standard deviation of repeatability that is obtained from the corresponding value of the repeatability limit $r$. According to [4], $r = 3\%$ of the average value of two repeated tests.

According to the ISO 5725-6:2005, $\sigma_r = \frac{|y_1 - y_2|}{2.8}$. So, $u_r = \sigma_r = \frac{r}{2.8}$.

Measurement uncertainty of correctness $\delta_d$. The value of the method correctness $B$ can be determined by: the systematic error of the balance, the competent personnel, incorrect setting of the automatic counter, if it is used.

We do not take into account the component due to the personnel incompetence if:

1) the condition of insignificance of the difference between the results of specialists obtained during the verification of the method is fulfilled [10]. Otherwise, it is necessary to find out the reasons for inappropriate performance of the method and repeat the verification.

[Fig. 1. Cause and effect diagram for the estimation of the thousand-seed weight]
The evaluation of the measurement uncertainty of the thousand-seed weight in accredited testing laboratories

Table 1

<table>
<thead>
<tr>
<th>Input value</th>
<th>Uncertainty components</th>
<th>Symbol</th>
<th>Probability distribution</th>
<th>Type of evaluation</th>
<th>Recalculation formula</th>
<th>Sensitivity coefficients $c_i$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\delta m_1$</td>
<td>Uncertainty of weighing the first sample</td>
<td>$u_m$</td>
<td>Gaussian</td>
<td>B</td>
<td>—</td>
<td>1</td>
</tr>
<tr>
<td>$\delta m_2$</td>
<td>Uncertainty of weighing the second sample</td>
<td>$u_m$</td>
<td>Gaussian</td>
<td>B</td>
<td>—</td>
<td>1</td>
</tr>
<tr>
<td>$\delta_h$</td>
<td>Repeatability</td>
<td>$u_r$</td>
<td>Gaussian</td>
<td>B</td>
<td>$u_r = \frac{r \cdot (m_1 + m_2)}{2 \cdot 2.8 \cdot 100}$</td>
<td>1</td>
</tr>
<tr>
<td>$\delta_d$</td>
<td>Rounding up the final result</td>
<td>$u_d$</td>
<td>Uniform</td>
<td>B</td>
<td>$0.01/2\sqrt{3}$ or $0.1/2\sqrt{3}$</td>
<td>1</td>
</tr>
</tbody>
</table>

2) the laboratory has a procedure and keeps records for supervising the personnel and monitoring their competence (direct requirements [2]).

The components arising from the systematic error of the balance and incorrect setting of the seed counter do not need to be considered as:

- we have the evidence that the equipment meets the requirements;
- the balances are calibrated in time, the obtained values of the calibration uncertainty at the operating points meet the quality requirements for the equipment;
- the intermediate checks to maintain confidence in the performance of the equipment are carried out: every day, on the use day of the balances, we carry out an intermediate check using a calibrated weight;
- the seed counter is checked with pre-prepared crop samples of 500 seeds each;
- we document and retain the results.

Rounding up the uncertainty $\delta_d$. Since the standard [4] provides rounding up of results, we add the rounding up component to the combined standard measurement uncertainty. The rounding up component is equal to $0.1/2\sqrt{3}$, and for small-seeded crops this component is $0.01/2\sqrt{3}$.

4.2. The uncertainty budget

As a result of the analysis (1), we have the following uncertainty budget, given in Table 1.

In general, we calculate the combined standard measurement uncertainty using the formula:

$$u_c(M) = \sqrt{\left(2 \cdot c_m \cdot u_m^2 + c_r \cdot u_r^2 + u_d^2\right)}.$$

The components are considered uncorrelated. The sensitivity coefficients are $c_r = 1$, $c_m = 1$.

Thus, the resulting formula for combined standard measurement uncertainty with the recalulation of the components will be as follows:

$$u_c(M) = \sqrt{\left(2 \cdot u_m^2 + \left(\frac{r \cdot (m_1 + m_2)}{2 \cdot 2.8 \cdot 100}\right)^2 + u_d^2\right)}.$$  \hfill (3)

The expanded uncertainty for testing the thousand-seed weight for a level of confidence 95% is calculated as:

$$U(M) = 2 \cdot u_c(M).$$

4.3. An example of evaluating the uncertainty of the thousand-seed weight

The method was tested for a sample of corn seeds of DKS CH (F1) cultivar produced in 2022. The sample was sent to be tested for certification to an accredited seed laboratory.

After having tested the “purity” indicator, two replicates of 500 seeds each were deducted from the main crop seeds without selection and weighed on the balance. The results were $m_1 = 180.97$ g for the first replicate and $m_2 = 182.11$ g for the second replicate.

The mean of the weights of both replicates is 181.54 g. The actual difference between the readings is 1.14 g, which does not exceed 3% of the mean of 5.45 g. Thus, we consider the analysis valid.

The weighing was carried out on laboratory electronic balance TBE-3-0.1-a, the standard measurement uncertainty of mass weighting on which, according to the internal regulatory document of the laboratory, is $u_m = 0.31$ g. The sensitivity coefficient is $c_m = 1$. The uncertainty component of the repeatability is $u_r = 1.966$ g. The uncertainty component of rounding up is $u_d = 0.029$ g.

The uncertainty budget for the case under consideration is shown in Table 2.

The combined measurement uncertainty (3) is

$$u_c(M) = \sqrt{\left(2 \cdot (0.31)^2 + (1.966)^2 + (0.029)^2\right)} = 2.014 \text{ g}.$$

The expanded measurement uncertainty is $U(M) = 4.0 \text{ g}$.

The analysis of the obtained estimates shows that the main contribution to the measurement uncertainty when applying the “two 500-seed samples” approach regulated by the standard [4] is the repeatability, and the other components can be neglected. The values of the remaining components meet the criterion of
Оцінювання невизначеності вимірювання маси тисячі насінин в акредитованих насінневих випробувальних лабораторіях

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Анотація
Оцінювання невизначеності вимірювання показників якості насінневого матеріалу є невід’ємною складовою проведення випробувань в акредитованих насінневих лабораторіях, залучених до процесу сертифікації насіння. Границі значення маси 1000 насінин не нормуються під час сертифікації, проте беруться до уваги під час визначення вартості партії та визначення норм висіву насіння, що свідчить про актуальність проведених досліджень.

У роботі подано результати розробленої методики оцінювання невизначеності вимірювання маси 1000 насінин з урахуванням всіх впливальних факторів. Розроблені основні розрахункові формули, залежні від специфіки випробувань, а також узагальнені результати випробувань з використанням даних по 5 насінницях, що сприяють уточненню результатів.

Видання з відомством: Український метрологічний журнал, 2023, № 1, 67–72

The uncertainty budget for the indicator of the thousand-seed weight

<table>
<thead>
<tr>
<th>Input value, g</th>
<th>Estimation of the input value, g</th>
<th>Standard uncertainty, g</th>
<th>Probability distribution</th>
<th>Type of evaluation</th>
<th>Sensitivity coefficients ( c_i )</th>
<th>Uncertainty contribution</th>
</tr>
</thead>
<tbody>
<tr>
<td>( m_1 )</td>
<td>180.97</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>( m_2 )</td>
<td>182.11</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>( \delta m_1 )</td>
<td>0</td>
<td>0.31</td>
<td>Gaussian</td>
<td>B</td>
<td>1</td>
<td>0.31</td>
</tr>
<tr>
<td>( \delta m_2 )</td>
<td>0</td>
<td>0.31</td>
<td>Gaussian</td>
<td>B</td>
<td>1</td>
<td>0.31</td>
</tr>
<tr>
<td>( \delta g )</td>
<td>0</td>
<td>1.966</td>
<td>Gaussian</td>
<td>B</td>
<td>1</td>
<td>1.966</td>
</tr>
<tr>
<td>( M_0 )</td>
<td>363.1</td>
<td></td>
<td>Uniform</td>
<td>B</td>
<td>1</td>
<td>0.029</td>
</tr>
</tbody>
</table>

The basic calculation formula for testing by using the method of two weights of 500 seeds each is determined. The impact of the repeatability and correctness of the measurement uncertainty is assessed. It is shown under what conditions the components of repeatability and correctness can be neglected in the combined measurement uncertainty. A formula for calculating the combined measurement uncertainty, taking into account all influencing variables, is presented. The calculation of the expanded measurement uncertainty when testing corn seeds received to be tested for the purpose of certification of the sowing qualities is presented.
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References
1. On approval of the procedure for certification, issuance and cancellation of certificates for seeds and/or planting material and forms of certificates for seeds and/or planting material: Cabinet of Ministers of Ukraine Resolution №97 from 21 February 2017. Update date: 11.11.2022 (date of application: 04.12.2022) (in Ukrainian). Available at: https://zakon.rada.gov.ua/laws/show/97-2017-%D0%BF#Text