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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.

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1. Introduction

The seed quality is one of the factors of high yielding crops. The production and circulation of seeds in Ukraine falls under the state supervision and is regulated by law [1]. Sowing qualities of the seed are a set of indicators that determine the suitability of seeds for sowing. The Resolution of Cabinet of Ministers of Ukraine [1] establishes a list of the seed sowing qualities. These qualities include colour, odour, purity and impurity, content of other species of the seed, germination, germinative energy, thousand-seed weight, diseases and pest infestation. The seed quality indicators are tested exclusively by seed testing laboratories that are accredited to meet the requirements of the standard [2]. The evaluation of the measurement uncertainty and all its major components is a fundamental requirement of the standard [2], non-compliance with the requirement of which leads to a significant nonconformity.

Ukraine also recognizes foreign seed certificates. The main requirements for the recognition of foreign seed certificates are:

- an agreement on mutual recognition of certification results shall be concluded with the certification body of the country of the seed origin;
- the products that are imported to Ukraine can be identified by accompanying documentation as being manufactured in accordance with the regulatory documents that are in force in Ukraine;
- the fundamental requirements and norms that are specified in foreign seed certificates shall be compatible with the corresponding Ukrainian requirements and norms.

Most of the certificates that accompany foreign lots of seeds do not contain all the indicators specified in [1]. According to the procedure approved by [1], samples must be taken from each lot, and additional tests must be conducted for the missing indicators. Among others, it is the thousand-seed weight. Test results must be accompanied by the measurement uncertainty. Limit values for the thousand-seed weight are not standardized during the certification process, but are taken into account when determining the lot cost and seeding rates. Therefore, it is important to develop a method for evaluating the measurement uncertainty of the seed quality indicators.

2. The analysis of literature sources and problem statement

General requirements for the evaluation of measurement uncertainty, as recommended in [2], are given in the ISO/IEC Guide 98-3 [3]. Methods for testing the seed quality indicators for certification purposes are regulated and described in [4]. Testing

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of the seed quality indicators 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.

$$M = m_1 + m_2, \quad (1)$$

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:

$$M_x = M + \delta m_1 + \delta m_2 + \delta_\xi + \delta_B + \delta_d, \quad (2)$$

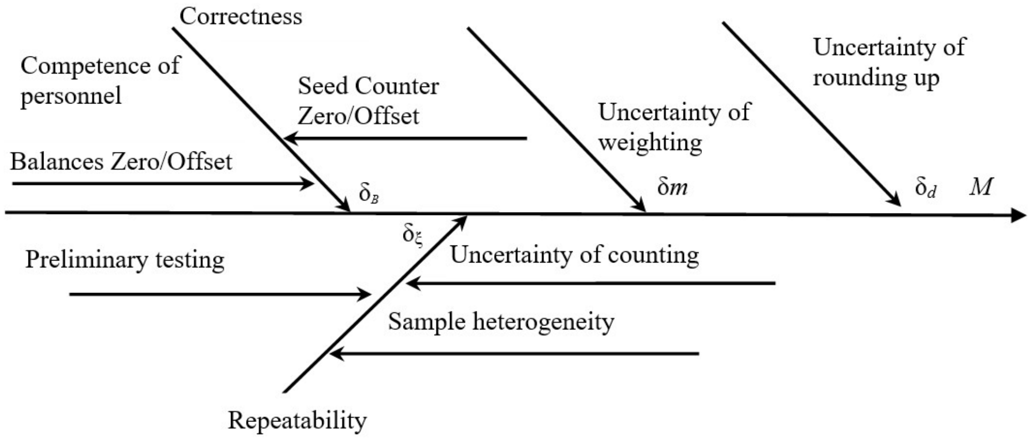

Fig. 1. Cause and effect diagram for the estimation of the thousand-seed weight

where δm_1 and δm_2 are the components of weighing measurement uncertainty, g; δ_ξ is a random component of the result, g; δ_B is a component of the correctness of the method, g; δ_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 $\delta m_1 = \delta m_2$. Given this, we denote the standard weighing measurement uncertainty as $u_m, (u_{m1} = u_{m2} = u_m)$.

Random components of the measurement uncertainty δ_ξ. 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 ξ 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 $s_A = s \cdot \sqrt{\frac{n-1}{n-3}}$, 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 δ_B. 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;

Table 1

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

Input value	Uncertainty components	Symbol	Probability distribution	Type of evaluation	Recalculation formula	Sensitivity coefficients c_i
δm_1	Uncertainty of weighing the first sample	u_m	Gaussian	B	–	1
δm_2	Uncertainty of weighing the second sample	u_m	Gaussian	B	–	1
δ_ξ	Repeatability	u_r	Gaussian	B	$u_r = \frac{r \cdot (m_1 + m_2)}{2 \cdot 2.8 \cdot 100}$	1
δ_d	Rounding up the final result	u_d	Uniform	B	$0.01/2\sqrt{3}$ or $0.1/2\sqrt{3}$	1

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 δ_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:

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

The components are considered uncorrelated. The sensitivity coefficients are $c_\xi = 1$, $c_m = 1$.

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

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

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_ξ = 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

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

The expanded measurement uncertainty is $U(M)$ = = 4.0 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

Table 2

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

Input value, g	Estimation of the input value, g	Standard uncertainty, g	Probability distribution	Type of evaluation	Sensitivity coefficients c_i	Uncertainty contribution
m_1	180.97					
m_2	182.11					
δm_1	0	0.31	Gaussian	B	1	0.31
δm_2	0	0.31	Gaussian	B	1	0.31
δ_ξ	0	1.966	Gaussian	B	1	1.966
δ_d	0	0.029	Uniform	B	1	0.029
M_x	363.1					4.0

insignificance: their sum is three times smaller than the repeatability component. It is possible to increase the accuracy of the expanded measurement uncertainty evaluation by increasing the number of working samples (weights) from the sample (four or more), which will allow obtaining a valid estimate of the repeatability. In this case, such a requirement should be documented in accordance with the requirements of clause 7.1 [2], and testing laboratories should use different methods for evaluating the measurement uncertainty.

5. Conclusion

The article analyses the most commonly used in the routine laboratory activities method of estimating the thousand-seed weight, which is used to determine the quality indicators of agricultural seeds for certification purposes and is regulated by the standard. 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.

Оцінювання невизначеності вимірювання маси тисячі насінин в акредитованих насіннєвих випробувальних лабораторіях

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Анотація

Оцінювання невизначеності вимірювання показників якості насіннєвого матеріалу є невіддільною складовою проведення випробувань в акредитованих насіннєвих лабораторіях, залучених до процесу сертифікації насіння. Граничні 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 стандартизованим методом двох наважок по 500 насінин. Для стандартизованого методу отримано основну розрахункову формулу. Визначено, що складову від повторюваності доцільно оцінювати за типом В, тому що наявні лише два результати випробувань. Цю складову необхідно брати як граничне значення фактичної розбіжності між двома повторами. Показано, що складові невизначеності, спричинені неоднорідністю зразка, невизначеністю підрахунку та впливом попередніх випробувань, можна не враховувати, якщо виконувати вимоги стосовно моніторингу компетентності персоналу та підготовки проб. Складову невизначеності від правильності методу можна не враховувати, якщо виконується умова незначущої різниці між результатами фахівців. Наведено формулу для оцінювання стандартної невизначеності. Основним джерелом невизначеності (інструментальної складової) є ваги. Методику апробовано в акредитованій насіннєвій випробувальній лабораторії. Наведено приклад розрахунку невизначеності вимірювання маси 1000 насінин від партії насіння кондиційної кукурудзи сорту, поданої на сертифікацію.

Ключові слова: показники якості насіння; маса 1000 насінин; сертифікація посівних якостей; невизначеність; акредитована насіннєва лабораторія.

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