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DETERMINATION OF METROLOGICAL TRACEABILITY INDICATORS IN THE ASSESSMENT OF HUMIDITY PARAMETERS

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ANNOTATION: In this article was given the informations on metrological traceability, its provision and determination of the total standard uncertainty in the testing process. Determination of errors in metrological traceability measurements for the method of "obtaining a test result directly from the indicator of measuring instruments" using the example of determining and measuring the moisture content of grain and grain products and provides recommendations for determining the total standard uncertainty index in the testing process.
Keywords: Metrology, measurement, testing laboratory, metrological traceability, DSt ISO/IEC 17025:2019, calibration, uncertainty, feed, standard uncertainty.

INTRODUCTION. At the current stage of development, product quality, safety and competitiveness are becoming an important factor in the rapid and sustainable development of economic sectors, increasing the profitability and efficiency of production. The stated goals cannot be achieved without ensuring the accuracy, objectivity, reliability, and comparability of measurement results used in various sectors of the economy and public administration. As part of the implementation of projects to modernize production and technically and technologically upgrade it, enterprises of the republic are working to introduce modern equipment into the technological process, including measuring instruments that control the quality and quantity of manufactured products. Given the trend of increasing the number and new types of measuring equipment in our country, the improvement of metrological activity and its infrastructure must constantly adapt to economic changes in Uzbekistan and the most favorable conditions for the development of local production, especially small businesses and private entrepreneurship [1].

Today, everything used in life has a precise size and there are devices to measure them. Taking measurements correctly is an important part of accurate calculations. To determine the true value of measurements, a measuring instrument must display accurate and correct values.

The provision of metrology services in our republic is developing year by year. In recent years, special attention has been paid to the development of the field of metrology in our country. In particular, over the past 5 years, a number of decrees and decisions have been adopted and roadmaps have been developed at the level of the head of state and the government. The new edition of the main legal document, the Law "About Metrology", was approved on April 7, 2020. Practical reforms carried out on the basis of this law and sub-legal documents are showing their results.

Today, reforms to ensure the accuracy of measurements include the task of accrediting testing laboratories. Accreditation of testing laboratories is carried out based on the Uzbek DSt ISO/IEC 17025:2019 standard "Requirements for the competence of testing and calibration laboratories". The metrological traceability parameter is important in this process.

Metrological traceability is the property of a measurement result to be traceable to a standard through a documented series of continuous calibrations [2]. The laboratory shall establish and maintain the metrological origin of its measurement results by conducting documented continuous calibrations, each calibration contributing to the uncertainty of the measurements and relating them to an appropriate standard. ISO/IEC Guide 99 defines metrological origin as “a property of a measurement result that can be compared with a reference value through documented continuous calibration, each calibration contributing to the uncertainty of the measurement” [3].

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Metrological origin is determined taking into account and ensuring:

a) determine the quantity to be measured;

b) a documented and defined chain of continuous calibrations against relevant standards (relevant standards include national and international standards as well as own standards);

c) assessing measurement uncertainty at each step of the measurement uncertainty chain according to agreed methods;

d) perform each step of the measurement chain in accordance with the relevant methods, measurement results and their associated recorded measurement uncertainties;

e) provide proof technical competence of laboratories that can perform one or more steps in the chain of control.

The systematic error (sometimes called "bias") of a measurement performed on calibrated equipment is taken into account to apply metrological origin to measurements performed by a laboratory. There are several mechanisms that allow for systematic errors in measurements to be taken into account when implementing metrological traceability.

Sometimes a standard containing data from a competent laboratory are used for the purpose of implementing the determination of metrological reference valuesThis information only includes a statement of compliance with the specification. This approach, where the specified boundaries are considered a source of uncertainty:

- using an appropriate decision rule to determine compliance;
- defined limits that are appropriately technically accounted for in the uncertainty budget.

The technical justification for the above approaches is that they are compliant with an approved specification, which defines the range of values to be measured. The true value lies within this range with a certain degree of accuracy, and this degree takes into account both any deviation from the true value and the measurement uncertainty [3]. Also, in accordance with the requirements of the GUM Guide [7] and the O'z DSt ISO/IEC 17025:2019, the assessment of measurement uncertainty must be indicated in the test reports [3].

MATERIALS AND METHODS. In this article we take the moisture measurement indicator for grains and grain products. Determining the moisture index of grain products O'z DSt 3121:2016 " Grain and grain products. Infrared thermogravimetric method for determination of moisture" is carried out in accordance with paragraph 9 of the state standard [4]. This is based on the methodology of "obtaining the test result directly from the indicator of the measuring instrument", that is, by obtaining the value of the quantity displayed on the screen (display) of the measuring instrument (Figure 1).

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Figure 1. Thermogravimetric infrared moisture detection equipment

A 5.0 g sample is initially taken for determine grain moisture content and the sample is placed in an infrared drying device and dried for 20 minutes. After that, the weight of the dried sample is determined (table 1). The weight loss (moisture content) is determined using the following formula (1) based on the determined weight data. In this way, the moisture loss in the grain sample before and after drying each measurement is determined, and this indicator is calculated in units of W percent (%).

$$W(\%) = \frac{m_1 - m_2}{m_1} * 100 \qquad (1)$$

here: m_1 – sample weight equal to 5.00 g;
m_2 – weight of a 5.00 g sample after drying, g;

The uncertainty of the results obtained through measurements should be assessed in accordance with clause 7.6.3 of the O'z Dst ISO/IEC 17025:2019 standard. The expanded uncertainty of the measurement results **U** is calculated using the following formula:

$$= \cdot U_c \qquad (2)$$

here: k – coverage ratio
U_c – total standard uncertainty in the test process

The expanded uncertainty of the measurement results is found by multiplying the standard uncertainty of the output quantity **U** by the coverage factor **k**. The value of the coverage coefficient **k** is taken depending on the level of measurement reliability. In most cases, the confidence level is assumed to be **k=1** for a 68% confidence interval, **k=2** for a 95% confidence interval, and **k=3** for a 99% confidence interval.

After all components of measurement uncertainty are determined, their total standard uncertainty U_c is estimated according to the law of uncertainty distribution [6]. The total standard uncertainty U_c in the test process is calculated using the following formula.

$$U_c = \sqrt{U_A^2 + U_B^2} \qquad (3)$$

here:

U_A– is the standard uncertainty of input quantities of type A. This type of uncertainty is taken into account if the number of measurements is more than 3, otherwise it is equal to 0.

U_B– this is the standard uncertainty indicator for type B, which is indicated on the calibration certificate of the measuring instrument.

RESULTS. The weight of the dried sample is determined and this results was given in following table 1.

Indicators obtained from the test results

table 1

№	Weight of the initial sample, m_1	Weight of dried sample, m_2	Determined humidity indicator, W (%)
1	5,0	4,81	3,8
2	5,0	4,85	3,0
3	5,0	4,85	3,0

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4	5,0	4,8	4,0
5	5,0	4,85	3,0
6	5,0	4,81	3,8
7	5,0	4,84	3,2
8	5,0	4,82	3,6
9	5,0	4,83	3,4
10	5,0	4,85	3,0

The results were processed to determine the uncertainty and the following indicators were obtained (Table 2).

Indicators obtained by processing measurement results

Table 2

W_i	X_i	$X_{o'r}$	$X_i - X_{o'r}$	$(X_i - X_{o'r})^2$	U_A	U_B	U_c	U
X_1	3,80	3,38	0,42	0,1764	0,1245	0,02	0,1261	0,28
X_2	3,00		-0,38	0,1444				
X_3	3,00		-0,38	0,1444				
X_4	4,00		0,62	0,3844				
X_5	3,00		-0,38	0,1444				
X_6	3,80		0,42	0,1764				
X_7	3,20		-0,18	0,0324				
X_8	3,60		0,22	0,0484				
X_9	3,40		0,02	0,0004				
X_{10}	3,00		-0,38	0,1444				

$$U_A = \sqrt{\frac{\sum (x_i - x_{o'r})^2}{n * (n-1)}} = \sqrt{\frac{1,396}{10 * (10-1)}} = 0,1245$$

U_B – The standard uncertainty for type B is given in the calibration certificate of the measuring instrument, and the calibration certificate of the thermogravimetric infrared moisture detector gives a standard uncertainty of 0,02.

$$U_c = \sqrt{U_A^2 + U_B^2} = \sqrt{0,1245^2 + 0,02^2} = 0,1261$$

The value of the coverage coefficient **k** is a coefficient that depends on the level of measurement reliability and the number of tests, and is determined from table 3.

The value of the coverage coefficient k

Table 3.

Number of tests	Reliability level		Number of tests	Reliability level	
	0,95	0,99		0,95	0,99
1	12,706	63,657	**11**	2,201	3,106

2	4,303	9,925
3	3,182	5,841
4	2,776	4,604
5	2,571	4,032
6	2,447	3,707
7	2,365	3,499
8	2,306	3,355
9	2,262	3,250
10	2,228	3,169
15	2,131	2,947
17	2,110	2,898
19	2,093	2,861
21	2,080	2,831
25	2,060	2,787
30	2,042	2,750
60	2,000	2,660
120	1,980	2,617
∞	1,960	2,576

Taking into account that the value of the coverage coefficient **k** is taken depending on the level of reliability of the measurement, since the number of tests was 10 and the level of reliability was within the 95% interval, **k=2.228** was taken and the expanded uncertainty indicator was determined as follows:

$$U = \quad \cdot U_c = 2{,}228 \cdot 0{,}1261 = 0{,}28$$

DISCUSSION. The uncertainty of the results of measuring the moisture content of grain products using a thermogravimetric infrared moisture analyzer was assessed in accordance with the requirements of the O'z DSt ISO/IEC 17025:2019 standard and was expressed as follows.

$$W=(3{,}38 \pm 0{,}28)\ \%$$

CONCLUSION. Thus, according to clause 6.4.5 of the standard O'z Dst ISO/IEC 17025:2019 "General requirements for the competence of testing and calibration laboratories", the equipment used for measurement must provide the accuracy or uncertainty required to obtain a valid result [3]. Based on this requirement, it is recommended to use the above formulas to determine the assessment of measurement uncertainty depending on the test results for the methods of obtaining the readings of the measuring instrument.

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