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# Assessment and investigation of measurement uncertainty of standard samples of substances and materials in physicochemical measurements based on standard test methods

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**Abstract.** The article assessed and investigated the uncertainty of measurements of standard samples of substances and materials in physicochemical measurements based on standard test methods. A general approach to estimating the sources of uncertainty of standard samples is described. Uncertainties from heterogeneity of standard sample material, uncertainty from instability of standard sample characteristic values, uncertainty from method of setting reference value are investigated. Purity of substances is the main parameter that needs to be paid attention to when studying their properties. This is all the more important when it comes to using a pure substance as some approximation to the prototype reference value, since only this unit of seven main units in the International System of Units (SI system) does not have its own standard. In this sense, an important scientific task is the comprehensive study of pure substances for their practical use as benchmarks for comparison in metrological works and analytical research. The main and very important part of the measurement traceability system are materials with certified (certified) content of components and defined uncertainties of these values (standard samples of the approved type), which require pure substances certified at a higher - reference level, i.e. comparison standards.

## 1. Introduction

In metrological practice, standard samples are used during measurements in the process:

- verification, calibration, testing, metrological certification, calibration of measuring instruments;
- metrological certification of measurement methods;
- control of errors of measurement methods during their application in accordance with the algorithms established in them, as well as for other types of metrological control [1-4].



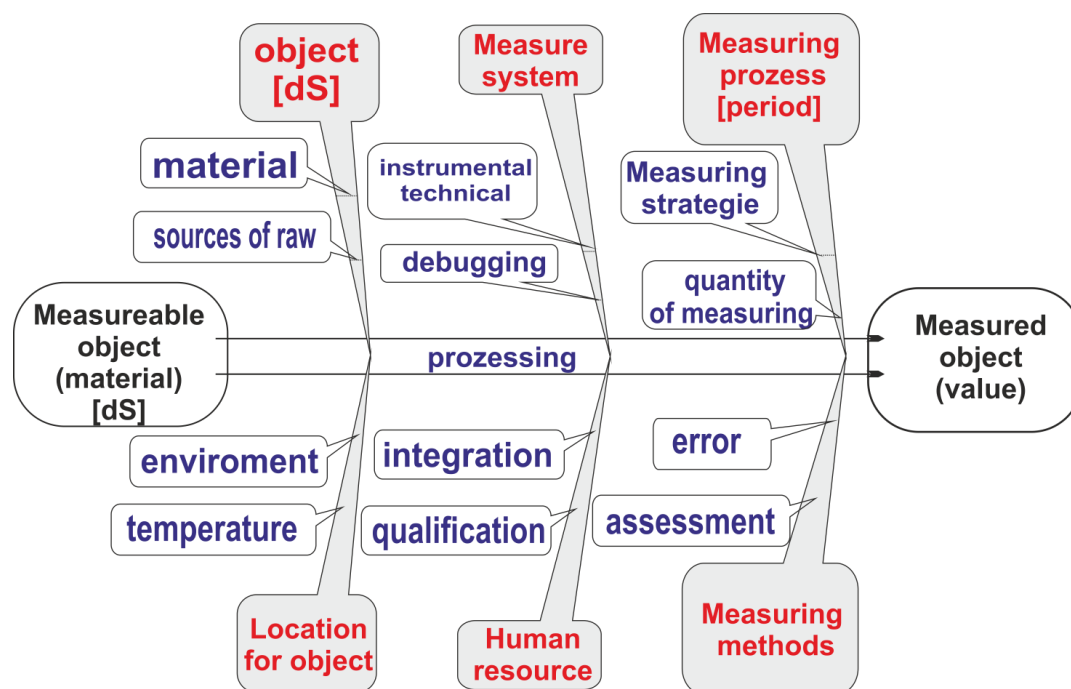
Currently, measurements related to the determination of the composition and properties of substances and materials are very widespread and play a responsible role in many areas of the national economy [5]. These are geophysical research, mining and enrichment, agricultural production, land reclamation and reclamation, environmental protection and monitoring, and health care. This includes the sanitary and epidemiological service, mining, coal, oil and gas, chemical, food industries, black and non-ferrous metallurgy [6]. At the same time, the objects of measurement are hundreds of thousands of substances and materials from these simple to the most complex biological objects, and the measured value can vary within  $(10^{-9}-99.99999) \%$  [7-10].

The variety of measurements given could not be based on unimaginable or spontaneous principles, since that would lead to unnecessary labour costs and low-cost expenditures and needed to be streamlined on a single scientific and methodological basis, ensuring the correctness and reliability of all the results obtained [11].

The essential specificity of measurement procedures, the difficulties associated with the storage and transfer of units of composition and properties of substances and materials, led to the need to apply special techniques and tools to solve these problems. The main, and often the only, means of storing and transferring unit sizes when measuring composition and, in some cases, properties of substances and materials are standard samples of composition and properties of substances and materials, which, as noted above, are measures.

## 2. Experimental Section

Figure 1 shows the main sources of measurement uncertainty when using reference materials as a reference.



**Figure 1.** Main sources and causes of measurement uncertainty of reference materials.

To characterize these sources in the regulatory documents, the following terms are used [12-16]:

- **method of certification of a reference material:** A metrologically substantiated procedure for establishing the certified value of a reference material.

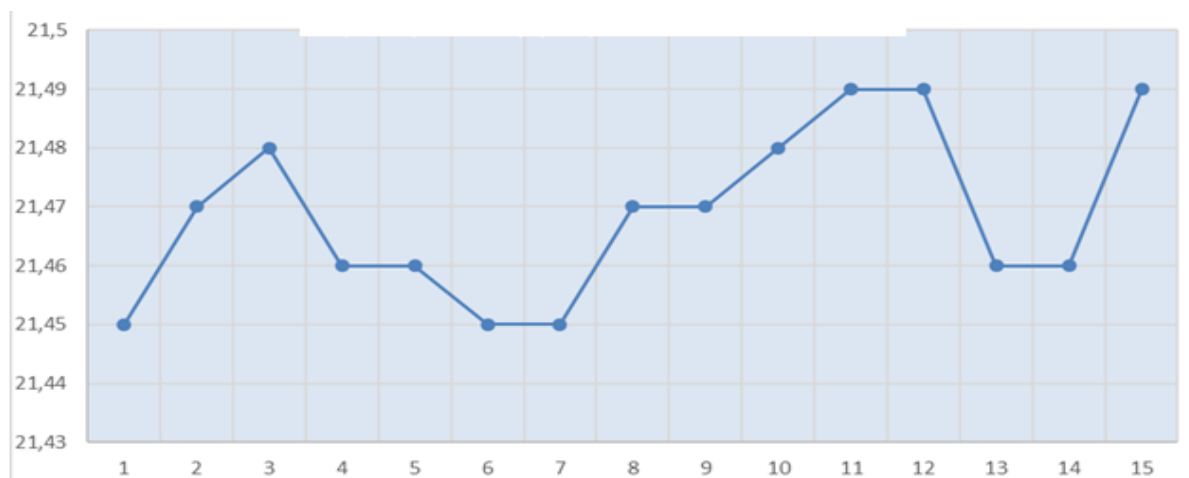
- **uncertainty of the CRM value:** A parameter characterizing the dispersion of values that could reasonably be attributed to the CRM characteristic.
- **standard uncertainty of the CRM value:** The uncertainty of the CRM value, expressed as a standard deviation.
- **expanded uncertainty of a CRM value:** The uncertainty of a CRM value, which is a quantity that defines the interval around the CRV value of a reference material within which, as can be expected, most of the distribution of values that could reasonably be attributed to the CRM characteristic sample.
- **uncertainty from the method of certification of a reference material:** The component of the uncertainty of the certified value of a reference material, due to the method of its certification.

In general, the total standard uncertainty of the certified value of reference materials is determined from the equation

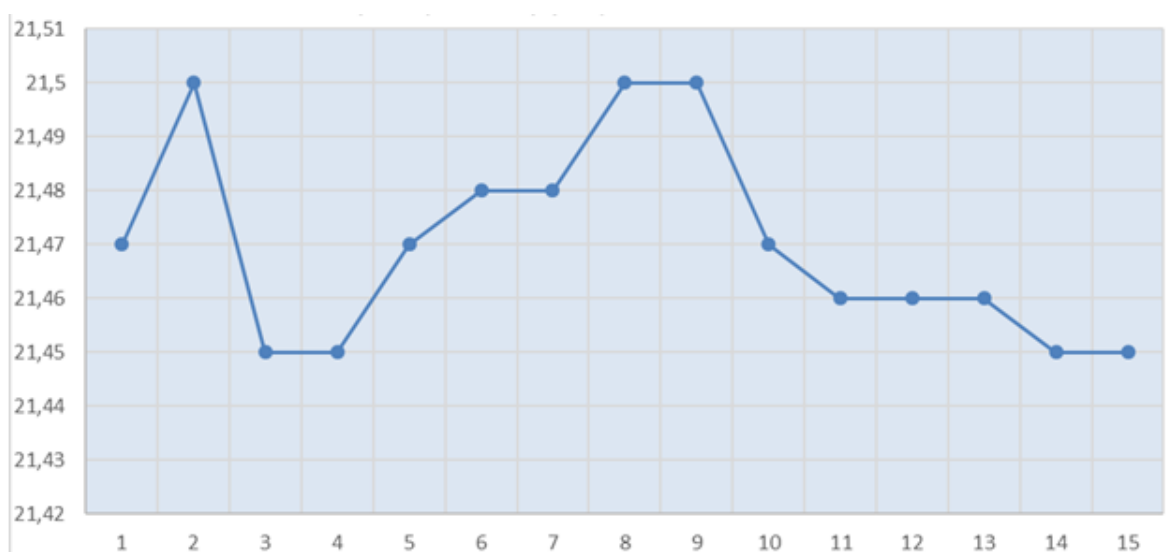
$$U_c^2(A) = U_{char}^2 + U_k^2 + U_{stab}^2 \quad (1)$$

Depending on the type of CRM material and storage conditions of CRMs, some components of the total standard uncertainty of the CRM certified value are either zero or their contribution to the CRM uncertainty may be insignificant.

Below is shown (figure 2, figure 3.) the scatter of the results of measurements and tests of food products, when standard images of substances and materials are used with an indication of the measurement uncertainty.



**Figure 2.** Spread and coefficient of variation of RSD of measurement results when using standard samples (1 experiment).



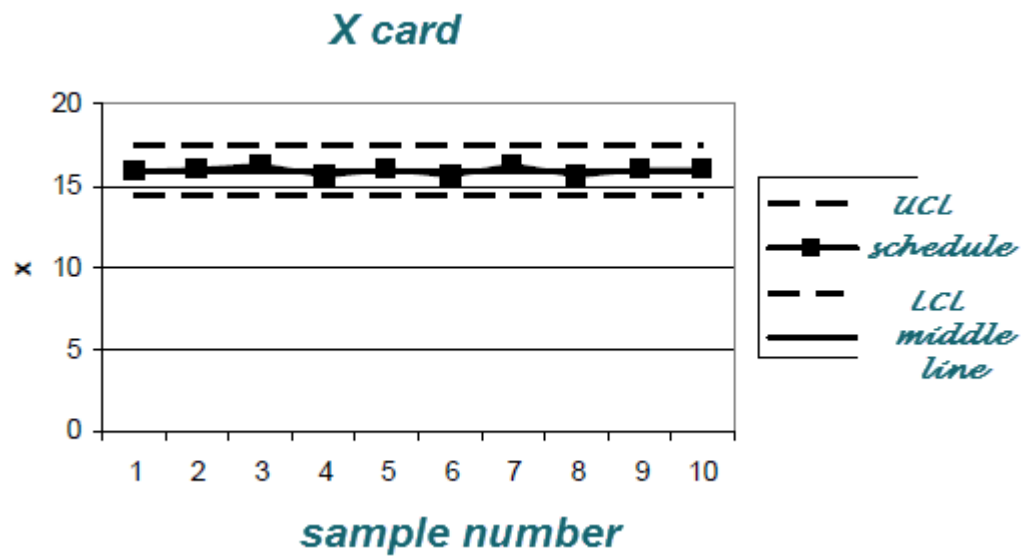
**Figure 3.** Spread and coefficient of variation of RSD of measurement results when using standard samples (2 experiment).

The table of values of the critical range coefficient  $f(n)$  is given in Table 1. Table 1.  $f(n)$  critical range coefficients

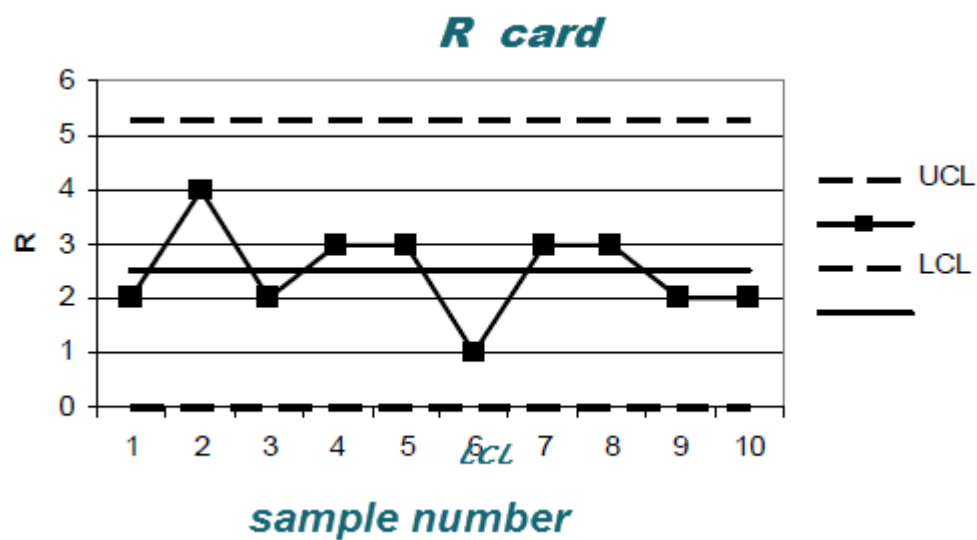
**Table 1.** Critical range coefficients.

<b>n</b>	2	3	<u>4</u>	5	6	7	8	9	10	11	12	13	14	15	16
<b>f(n)</b>	2.8	3.3	<u>3.6</u>	3.9	4.0	4.2	4.3	4.4	4.5	4.6	4.6	4.7	4.7	4.8	4.8
<b>n</b>	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31
<b>f(n)</b>	4.9	4.9	5.0	5.0	5.0	5.1	5.1	5.2	5.2	5.2	5.3	5.3	5.3	5.3	5.3
<b>n</b>	32	33	34	35	36	37	38	39	40	45	50	60	70	80	90
<b>f(n)</b>	5.3	5.4	5.4	5.4	5.4	5.4	5.5	5.5	5.5	5.6	5.6	5.8	5.9	5.9	6.0
<b>n</b>	100														
<b>f(n)</b>	6.1														

The X- and R- cards obtained as a result of calculations by the indicator. The acidity of raw milk is presented in figures 4 and 5. The statistics show that the use of reference materials with low standard uncertainties (U) provides verification criteria for food methods.



**Figure 4.** X-card in terms of acidity of raw milk.



**Figure 5.** R-card for the acidity index of raw milk.

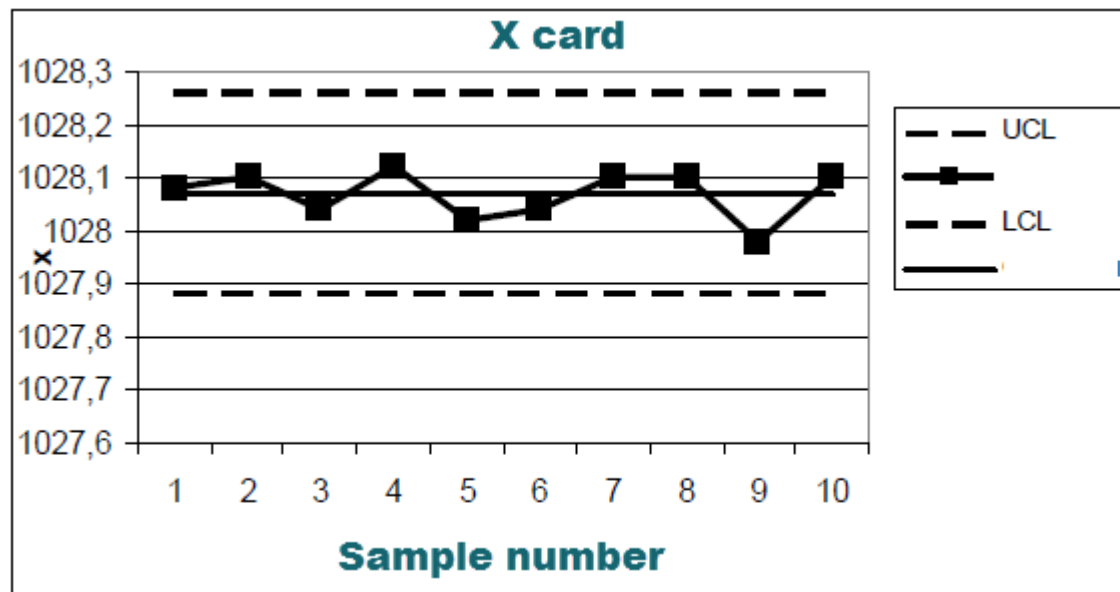


Figure 6. X-map in terms of density.

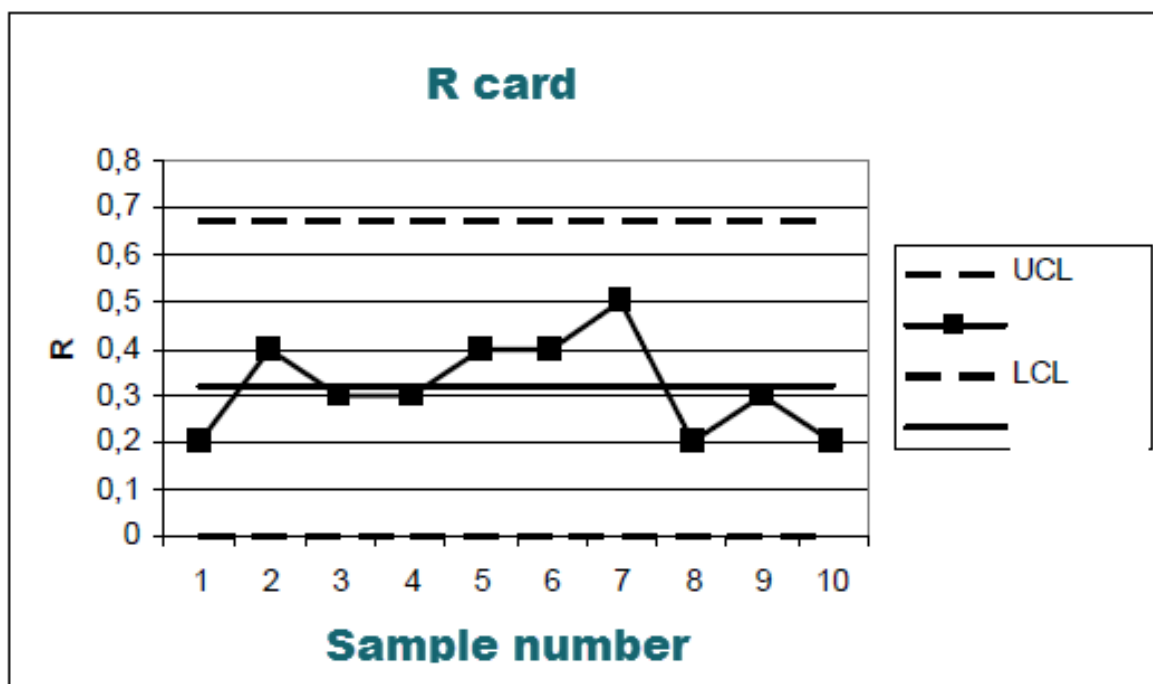


Figure 7. R- map according to the density index of raw milk.

### 3. Results and Discussion

In practice [17], the uniform distribution of random variables is mainly used to assess the uncertainty of reference materials. Because interstate standards and test methods indicate maximum permissible errors. Below we will see the main quantitative characteristics of this distribution law.

The measured value with probability 1 takes values in the interval  $[-a, +a]$  with the same probability for any part of the interval [18].

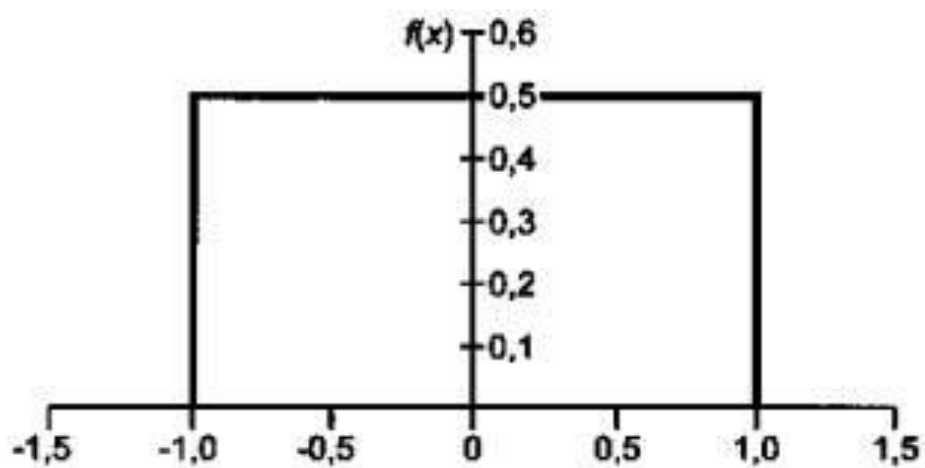
Measured value density function:

$$f(x) = \begin{cases} \frac{1}{2a}; & \text{for } -a \leq x \leq a \\ 0; & \text{for } |x| > a \end{cases} \quad (2)$$

Type B evaluation of measurement uncertainty:

$$u(x) = \frac{a}{\sqrt{3}} \quad (3)$$

The uniform distribution density function on the interval  $[-1, +1]$  is shown in figure 8:



**Figure 8.** Density function of uniform distribution on the interval  $[-1, +1]$ .

Application procedure:

- limits are given in the certificate or other accompanying documentation without indicating a confidence level, without any reason to believe that any part of the interval is more likely than another;
- the estimate is given in the form of the maximum range ( $\pm a$ ), and the shape of the distribution is unknown [19].

For example, before performing measurements, chromatographs are calibrated using a standard gas mixture and for further measurements the value of this standard gas mixture is considered a reference. The measurement results are analyzed on the basis of the interstate standard GOST 31371.7-2008 “Natural gas. Determination of the composition by gas chromatography with an uncertainty estimate. Part 7. Methods for making measurements of the molar fraction of components” and the measurement uncertainty is estimated. In this case, the standard gas mixture must have a certificate (Certified Reference Materials - CRMs) provided by the manufacturer that meets the requirements of the ISO 17034 standard [20].

#### 4. Summary



Standard samples play a key role in chemical and other types of measurements, ensuring their unity, comparability. Being one of the affordable and effective means transferring units of quantity, reference materials are widely used in millions of laboratories different countries to control the accuracy of measurement results, calibration, verification, calibration measuring instruments, validation of measurement techniques, in assessing the qualifications of laboratories and confirmation of the measuring capabilities of national metrology institutes. When carrying out tests using measuring instruments (tests), the metrological characteristics of which are readjusted before each measurement or regularly in short periods. Calibration of measuring instruments (tests), which require the establishment of a reference value using a standard sample or other measuring instruments (internal calibration - in-house calibration) before testing, can be carried out by the testing laboratory itself. For the full implementation of the international standard ISO / IEC 17025: 2017, reference materials are required that meet the requirements of ISO 17034.

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