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Verification of methods in accordance with the requirements of ISO/IEC 17025:2017 by the intralaboratory method

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Abstract

The article is dedicated to the analysis of the requirements of the standard ISO/IEC 17025:2017 for the verification of test and calibration methods. The necessity of verification of standardized methods is demonstrated and the characteristics of standardized methods that need to be confirmed during their verification are revealed. The method of intralaboratory verification of test methods is proposed, which is to confirm the repeatability and trueness of methods based on the results of control measurements of reference materials in accordance with the principles set out in the series of international standards ISO 5725. This method can be applied to calibrate measures of physical quantities.

In case of absence of reference materials, the verification of methods can be performed by interlaboratory comparisons. It is noted that the verification of test methods is a mandatory requirement for calculating the measurement uncertainty according to the reproducibility indicators given in the standardized method. The recommendations for laboratory actions to improve accuracy in case of unsatisfactory verification results are given.

Keywords: test method; verification; repeatability; trueness; uncertainty measurement.

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Introduction

An important factor in obtaining reliable test or calibration results is the availability of appropriate methods in the laboratory. ISO/IEC 17025:2017 [1], the national version of which is DSTU EN ISO/IEC 17025:2019 [2], in accordance with the requirements of which calibration and testing laboratories are accredited, contains virtually no restrictions on the choice of methods. It is recommended to choose methods published in international, regional or national standards, or published by reputable technical organizations, or published in relevant scientific literature or journals, or those specified by manufacturers in the operating documentation for the equipment. However, the laboratory may develop its own method or modify techniques for future use if the above conditions are missing or for some reason do not suit the laboratory.

It should be noted that in some cases national legislation restricts the freedom to choose the method, because in accordance with Article 7 of the Law of Ukraine "On metrology and metrological activity" [3] measurement methods in the field of statutory metrology, which are mandatory-legal acts or in normative documents to which there are corresponding references in normative-legal acts. Activities related to the field of legally regulated metrology are defined in Article 3 of the Law of Ukraine [3], in particular, it includes medical science, quality control, safety of food and medicines, environmental protection, etc.

However, no method, even the best one, can provide reliable results if the laboratory performs it incorrectly. Therefore, [1] requires that "the laboratory shall verify that it can properly perform methods before introducing them by ensuring that it can achieve the required performance". Since the latest version of ISO/IEC 17025:2017 [1], unlike the previous one [4], where this requirement is applied only to standardized methods, does not specify which methods should be verified – all methods are subject to verification. Thus, at first glance, the new version of ISO/IEC 17025 strengthens the requirements for verification of methods. In fact, if we take into account that all methods other than standardized must be validated, and, as defined in [1, 2], "validation is verification, where the specified requirements are adequate for an intended use", i.e. laboratory-validated methods

are considered already verified, it remains to verify only standardized methods. Practice shows that most of the requirements of ISO/IEC 17025:2017 for the verification of laboratory methods are validated.

It would seem that there is nothing wrong with this. Having validated standardized methods, the laboratory will better study them, understand their strengths and weaknesses, which will reduce the likelihood of inaccurate results. However, given the complexity of validation, this may require significant resource costs, including time, which with a large number of standardized accreditation methods can adversely affect the financial efficiency of the laboratory. The subject of this paper is to develop a method of verification of standardized methods by internal laboratory method.

Main part

According to [1], "verification is provision of objective evidence that a given item fulfils specified requirements". Despite the fact that today at the international level the accuracy of measurement results is assessed due to measurement uncertainty, most standardized methods, including international, specify indicators of repeatability, reproducibility and, rarely, trueness. However, this is equivalent to indicating the accuracy of the method, because, for example, according to [5, 6], the standard deviation of reproducibility is a good estimate of the total standard uncertainty. During the verification, the laboratory must demonstrate that its results are compatible with the requirements of the standardized method, and therefore must confirm that during the implementation of the method in the laboratory it achieves these indicators.

As is known [7, 8], repeatability is the degree of closeness to each other of the results of repeated measurements of the same value obtained under repeatability conditions, in other words, under the same conditions. This means that the measurement is performed by the same operator, at the same workplace, using the same equipment, at the same influential values and in the shortest possible time. Due to the presence of uncontrolled random influences or, in other words, due to the presence of a random measurement error, the results obtained under the conditions of obvious stability may differ from each other. According to [7], the convergence can be quantified by the standard deviation repeatability σ_r. Nevertheless, standardized methods often do not give the standard deviation repeatability, but the allowable difference r between the extreme (largest and smallest) results from n repeated measurements for a confidence level of $P = 0.95$. It can be converted to the standard deviation repeatability by the formula

$$\sigma_r = r/f(n),$$

where $f(n)$ is a coefficient, the value of which, depending on the number of repeated measurements n, are given in Table 1 [9].

Repeatability control is to obtain a series of m results in repeatability conditions. According to these results, the statistical estimate of the standard deviation of repeatability is calculated:

$$S_r = \sqrt{\frac{\sum (x_i - \bar{x})^2}{m-1}}.$$

According to [9], the repeatability of results in the laboratory is satisfactory if the condition [9] is met:

$$S_r^2/\sigma_r^2 \le \chi_P^2(\nu)/\nu,$$

where $\chi_P^2(\nu)$ is the distribution quantile χ^2 for the confidence interval $P = 0.95$ and the number of degrees of freedom $\nu = m-1$. The values of the quantiles are shown in Table 2.

For higher values of νthe values of the quantiles χ^2 can be found in statistical reference books.

Reproducibility is the degree of closeness to each other of the results of repeated measurements of the same value obtained in the conditions of reproducibility, i.e. in different laboratories, by different operators,

Table 1

The value of $f(n)$ depending on n for $P = 0.95$

n	2	3	4	5	6	7	8	9	10
$f(n)$	2.8	3.3	3.6	3.9	4.0	4.2	4.3	4.4	4.5

Table 2

The value of the distribution quantiles χ^2 for the confidence interval $P = 0.95$

ν	1	2	3	4	5	6	7	8	9	10
$\chi^2_{0.95}(\nu)$	3.8	6.0	7.8	9.5	11.1	12.6	14.1	15.5	16.9	18.3

on different equipment. Reproducibility can be quantified by the standard deviation reproducibility σ_R. However, the methods most often do not specify the standard deviation reproducibility, but the maximum allowable difference R between the results of the indicator in two different laboratories for a probability of 0.95. Then $\sigma_R = R/2.8$.

The standard deviations repeatability and reproducibility can be used to verify trueness. It should be recalled that according to [7] trueness is the degree of approximation of the average value from a large number of repeated measurements to the accepted reference value. The bias is a quantitative measurement of trueness, because the deviation of the average value from the accepted reference value is an estimate (approximate value) of a systematic error. If there is a certified reference material, in the certificate of which the assigned value μ is specified, which is taken as the reference value, the verification is reduced to obtaining m measurement results of the reference material. Based on these results, the average value of $\overline{x}$ is estimated and compared with the reference value of μ. The trueness of the measurement is satisfactory if the condition [9] is met:

$$\left|\overline{x} - \mu\right| \le 2 \cdot \sqrt{\sigma_R^2 - \sigma_r^2 \cdot \frac{m-1}{m}}.$$

In order to save resources, experiments to verify repeatability and trueness can be combined, i.e. the repeatability can be assessed based on the results of measurements of the reference material. It should be also noted that both during the repeatability check and during the trueness check, the experimentally obtained values should not be subjected to statistical processing to detect errors.

Regarding the number of m results to check the convergence and correctness, we can provide some general recommendations. It is recommended to get at least 10 results, but getting more than 20 results usually does not make sense, because if the number of results increases, the m gain in the accuracy of the estimated indicators is insignificant. However, if obtaining each result requires significant costs, this number may be less than 10.

The results of the verification of the method should be documented in the form of a report (protocol) on the verification with a detailed description of how the verification was performed. The date of verification, the facilities where the verification was performed, climatic factors affecting the results, involved personnel, obtained results together with all calculations must be at least documented. It is mandatory that the conclusion be drawn based on the verification results.

Repeated verification is performed provided the changes are made to the standardized method by the organization that standardized it.

In case of absence of reference materials, intralaboratory verification of correctness is a problematic task. A good solution to this problem can be an interlaboratory experiment, which implies interlaboratory comparisons or bilateral comparisons with a higher-level laboratory, but the consideration of this issue goes beyond this paper.

It should be noted that the verification performed in this way gives grounds for the evaluation of measurement uncertainty through standard deviation reproducibility, as [5, 6] require that the laboratory must first confirm that the quality of the test method performance meets the requirements specified for the method by verifying the correctness and convergence. This proves that the published data on the use of the method are consistent with the results of measurements and tests obtained by the laboratory. In this case, the expanded uncertainty U for the confidence level $p = 95\%$ can be estimated by the formula

$$U = 2 \cdot \sigma_R.$$

If the results of the method verification are unsatisfactory, it is necessary to carefully analyze the implementation of the method in the laboratory to identify excessive adverse effects. In particular, the reason for unsatisfactory repeatability may be excessive variability of some factors, such as excessive voltage instability in the power supply network of electronic instruments, fluctuations in external electromagnetic fields, vibration, and so on. Unsatisfactory trueness can be caused by hidden metrological failures of instrument, improper quality of reagents, incorrect systematic actions of the operator, excessive deviation of an influential factor from the normal value or an unfavorable combination of influential factors. In the case of exposure to such factors, measures should be taken to minimize or eliminate their impact. If all the measures taken failed, the characteristics of the method should be re-evaluated, that is, its validated.

All of the described above concerns mainly testing methods. The described approach can be partially applied to some calibration methods, in particular, methods for calibrating measures of physical quantities. Measures of physical quantities tend to have an inconsequential random error, i.e. convergence for the relevant techniques is a characteristic of the method rather than of the measure. An analogue of reference materials here can be a measure calibrated by a laboratory of higher metrological level with low uncertainty. To calibrate instruments with a significant random error, this approach cannot be implemented, since the observed variability of results during calibration of such instruments is due to the properties of the calibration object.

Finally, it should be noted that method verification does not exempt the laboratory from statistical control of the measurement procedure, including

regular correctness and convergence checks, and the application of quality assurance methods provided for by the laboratory management system, involving trained and qualified personnel. It is strongly recommended to use control cards [6].

Conclusions

The requirements of ISO/IEC 17025:2017 for the verification of test and calibration procedures have been analyzed. A method of verification of test methods confirming the convergence and correctness of the techniques in accordance with the principles set out in the series of international standards ISO 5725 is proposed. It is noted that the verification of methods is a mandatory requirement for calculating the measurement uncertainty by reproducibility indicators.

Верифікація методик відповідно до вимог ISO/IEC 17025:2017 внутрішньолабораторним способом

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Анотація

Статтю присвячено аналізу вимог стандарту ISO/IEC 17025:2017, національним аналогом якого є ДСТУ EN ISO/IEC 17025:2019, щодо верифікації методик випробувань та калібрування. Проаналізовано зміни у вимогах у порівнянні з попередньою версією вказаного стандарту. Зазначено особливості національного законодавства щодо вибору методик у лабораторіях у порівнянні з вимогами ISO/IEC 17025:2017. Констатується, що верифікація нестандартизованих методик зводиться до їх валідації. Показано необхідність верифікації стандартизованих методик та виявлено характеристики стандартизованих методик, що потребують підтвердження під час верифікації таких методик. Запропоновано спосіб внутрішньолабораторної верифікації методик випробувань, що полягає у підтвердженні збіжності та правильності методик на основі результатів контрольних вимірювань значень сертифікованих стандартних зразків відповідно до принципів, викладених у серії міжнародних стандартів ISO 5725. Цей спосіб верифікації може бути застосовано й до деяких методик калібрування, зокрема, методик калібрування мір фізичних величин. Показано незастосовність цього способу верифікації для методик калібрування приладів із суттєвою випадковою похибкою. У разі відсутності сертифікованих стандартних зразків верифікацію методик може бути проведено шляхом міжлабораторних порівнянь. Зазначено, що верифікація методик випробувань є необхідною умовою для розрахунку невизначеності вимірювання за показниками відтворюваності, наведеними у стандартизованій методиці. Виявлено фактори, які можуть бути причиною незадовільної верифікації методик, та наведено рекомендації щодо дій лабораторії для підвищення точності результатів вимірювань у цьому разі.

Ключові слова: методика випробування; верифікація; збіжність; правильність; невизначеність вимірювання.

Верификация методик в соответствии с требованиями ISO/IEC 17025:2017 внутрилабораторным способом

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Аннотация

Статья посвящена анализу требований стандарта ISO/IEC 17025:2017 по верификации методик испытаний и калибровки. Показана необходимость верификации стандартизованных методик и установлены их характеристики,

требующие подтверждения при верификации. Предложен способ внутрилабораторной верификации методик испытаний, заключающийся в подтверждении сходимости и правильности методик на основе результатов контрольных измерений значений стандартных образцов в соответствии с принципами, изложенными в серии международных стандартов ISO 5725. Этот способ может быть применен и к некоторым методикам калибровки, в частности, методикам калибровки мер физических величин. В случае отсутствия стандартных образцов верификация методик может быть проведена путем межлабораторных сравнений. Отмечено, что верификация методик испытаний является необходимым условием для расчета неопределенности измерений по показателям воспроизводимости, приведенным в стандартизованной методике. Приведены рекомендации по действиям лаборатории для повышения точности при неудовлетворительных результатах верификации.

Ключевые слова: методика испытания; верификация; повторяемость; правильность; неопределенность измерения.

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