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FEATURES OF THE PROCESSING OF RESULTS AND ESTIMATION OF MEASUREMENT UNCERTAINTY OF INTER-LABORATORY COMPARISON FOR CALIBRATION LABORATORIES

Inter-laboratory comparisons for calibration laboratories (CL) are carried out by competent providers. For assessment the results of CL participation in such tests used the criteria set by the test coordinators for the quality of calibration for a particular type of measurement shall be used. The main task of the provider is to establish the reference value of the measured value and its uncertainty. Inter-laboratory comparisons by the CL are based on three main types of test organization, where the coordinators – providers (reference laboratories) can be: National Metrology Institutes (NMI); accredited CL; accredited providers that are not NMI or accredited CL. To check the consistency of the results of inter-laboratory comparisons using the χ^2 criterion is proposed.

Keywords: *inter-laboratory comparison, calibration laboratory, uncertainty of measurements, transmission sample, National Metrology Institute.*

Introduction

Problem statement. Inter-laboratory comparisons (ILC) are one form of experimental verification of the work of laboratories to determine technical competence in a particular activity. The successful results of the ILC for the laboratory confirm competence in certain types of measurements by a particular specialist on specific equipment.

The ILC is the organization, performance and evaluation of calibrations/tests on the same or similar calibrations/tests items by two or more laboratories in accordance with predetermined conditions.

To obtain reliable results from ILC for accredited testing and calibration laboratories, it is necessary to improve the methods of processing their results. These methods are based on various data processing algorithms, therefore, it is necessary to select the most optimal method that would have the minimum number of application restrictions and allow receive objective data [1–2].

Unsatisfactory ILC results can be related not only to deviations from the normal state of the laboratory's competence, but also to equipment failures or lack of competence of the specialist who worked with it [3–4].

Of particular importance is the ILC for calibration laboratories (CL), which provide calibration of measuring instruments (MI) for accredited testing laboratories (TL). ILC programs should be prepared by competent providers (reference laboratories, RL). The main task of the RL ILC is to establish the reference (assigned) value of the measured value and its interval of uncertainty. The level of the results of the laboratories participating in the ILC depends on the competence and technical level of the RL.

The analysis of recent researches and publications. The ILC is an obligatory and integral part of the external quality control of the measurement results obtained in the overall CL quality system. The ILC plays an important role in evaluating CL's technical competence both during certification and in monitoring the activities of the laboratory during accreditation.

The ILC program for specific types of MI calibration is developed taking into account the requirements of ISO/IEC 17025 [5], EN ISO/IEC 17043 [6] and ISO 13528 [7]. According to the ISO/IEC 17025 standard, CL and TL in the accreditation field periodically conducted the ILC for confirmation competencies.

The number of publications devoted to the issues of organizing the ILC and the methods for processing the results in specific fields is increasing significantly.

A number of papers are devoted to the urgent questions of processing the data obtained in the ILC: in [8] two ways of processing ILC data was presented in the same procedure; [9] discusses the use of different methods for evaluating inconsistent data received; in [10] an application was proposed for processing the results of the ILC Bayesian approach; [11] suggested approaches to verifying the reliability of the measured measurement results in the ILC with the participation of CL; [12] reviewed software verification issues for the evaluation of ILC data; in [13] the application of *z* score test for evaluation of performance of CLs is recommended instead of E_n number since this number is not applicable due to the difficulty in determining the reference value.

Other works are devoted to the evaluation of the results of specific ILCs and their peculiarities: in [14], particular attention was paid to the quality control of ILCs carried out by accredited CLs, and the methods of analyzing the data obtained by the ILC model were considered; in [15–19] the issues of improving the methods for estimating the uncertainty of different types of measurements (pressure, flow rate, active power, temperature, electricity) for the ILC with participation of CL are considered; in [20–23], the main attention is paid to the evaluation of the CL data obtained in the ILC for specific types of measurements (reactive power, length, pressure, Vickers hardness) with peculiarities of estimating uncertainty of measurements; in [24–26], algorithms for conducting ILC and obtaining precision data for estimating gauge and measuring capabilities of laboratories are considered.

However, these publications do not take into account the drift of reference samples, which can significantly affect the ILC results from MI calibration, especially precision. In addition, in these publications there is no research at all about the required level of competence, including the technical, ILC providers.

Purpose of the article. The purpose of the article is to improve the method of processing primary data of ILC trials involving CL, based on three main types of test organization when the RL may be: National Metrology Institutes (NMI); accredited by CL; accredited providers (RLs) that are not NMI or accredited by CL. The relevance of such research stems from the growing need for ILC with the participation of CL to ensure the recognition of the results of product testing both at the national and regional levels and internationally. It is expedient to carry out a detailed assessment of uncertainty of measurements according to requirements [27–28]. An important element in ensuring the required level of reliability of the results is their verification of consistency.

Exposition of basic material

Metrological traceability requires an established calibration hierarchy. The general scheme of metrological traceability with using of calibration hierarchy is shown on fig. 1.

The technical basis of the Mutual Recognition Arrangement (MRA) of International Committee for Weights and Measures (CIPM) is the set of results obtained through key comparisons (KCs) of national measuring standards carried out by the Consultative Committees (CCs) of the CIPM and the Regional Metrology Organizations (RMOs). The RMOs organize corresponding RMO KCs with a number of common participants and with protocols allowing their results to be linked to those of the CC KC once these are treated in terms of equivalence [29].

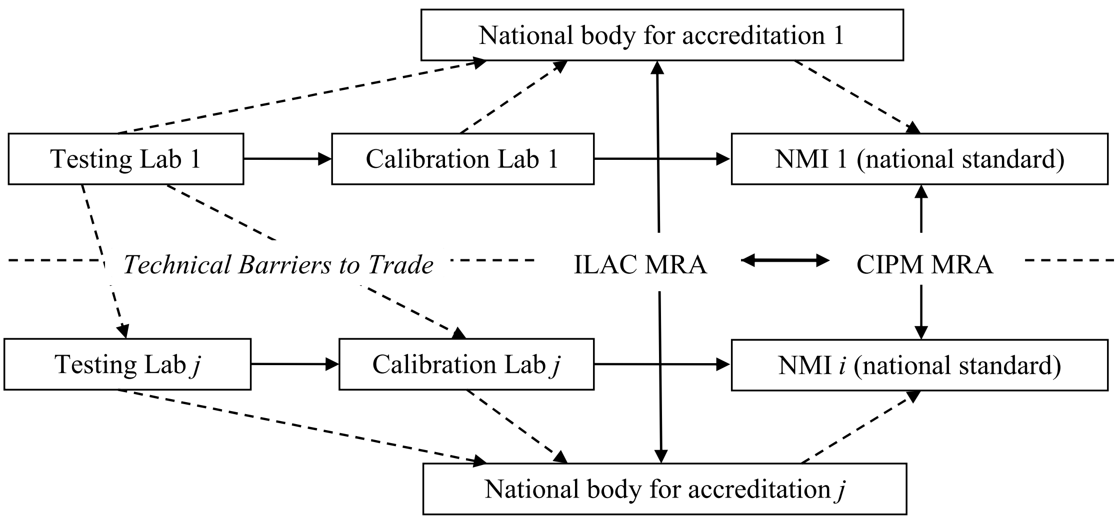

Fig. 1. General scheme of metrological traceability with using of calibration hierarchy

The CIPM MRA is the framework through which NMIs demonstrate the international equivalence of their measurement standards and the calibration and measurement certificates they issue. The outcomes of the CIPM MRA are the internationally recognized Calibration and Measurement Capabilities (CMCs) of the participating NMIs [30].

The International Laboratory Accreditation Cooperation (ILAC) considers the elements for confirming metrological traceability to be an unbroken metrological traceability chain to national measurement standard (NMIs), a documented measurement uncertainty, a documented measurement procedure, accredited technical competence, metrological traceability to the International System of Units (SI), and calibration intervals [31]. The ILAC MRA provides significant technical underpinning to the calibration and in turn delivers confidence in the acceptance of results.

According to ISO/IEC 17025 [5] CL shall have quality control procedures for monitoring the validity of calibrations undertaken. This monitoring may include the participation in inter-laboratory comparisons. By these means CL can provide evidence of its competence to its clients and the accreditation body [32].

ILCs for CLs are conducted in different countries within the framework of national accreditation bodies' activity. For ensure the mutual recognition of the calibration results, it is advisable to establish the relationship between these tests. To do this can use the results of international comparisons of national measuring standards stored in the NMI. In this case, the degrees of equivalence (DoE) of the NMI standards and its uncertainty may be taken into account [29]. Thus, it is possible to establish metrological traceability of CL standards to the corresponding national measuring standards [33–37].

The RL processing the data received from the CL participants according to the results of the ILC for CL. Requires verification of ILC data for consistency. In the case of uncoordinated data, an analysis is conducted for the purpose of rejecting this data or for further harmonization by correction the applied indicators. To verify the consistency of the data, comparative analyses of the relevant criteria for the performance statistics is carried out and select the most effective for use in the processing of the data [6–7].

Inter-laboratory differences for i-th CL traditionally defined by [6]:

$$D_{lab\ i} = x_{lab\ i} - X_{AV}, \tag{1}$$

where $x_{lab\ i}$ is measured value for i-th CL; X_{AV} is assigned value (AV) is determined by the RL.

Most often, the evaluation of data for the i-th CL is made using the E_n-index, which is determined by [6]:

$$E_n = \frac{x_{lab\ i} - X_{AV}}{\sqrt{U_{lab\ i}^2 + U_{AV}^2}}, \tag{2}$$

where $U_{lab\ i}$ is extended uncertainty of measurement of the value of the parameter of the comparison sample (a certain MI) by the CL participant; U_{AV} is extended uncertainty of measurements in determining the true value of the parameter of the MI.

The extended uncertainty of U_{AV} measurements is determined by:

$$U_{AV} = k\sqrt{u^2(X_{AV}) + u^2(X_{stab})}, \tag{3}$$

where k is coverage factor (traditionally k=2); $u(X_{AV})$ is standard uncertainty obtained during the calibration of the MI of RL; $u(X_{stab})$ is the standard uncertainty about the instability of the MI during the ILC.

The general algorithm for processing the received ILC primary data is described in [4]. This algorithm allows the RL to take into account all the reporting features of the ILC.

Linking the correspondingly extended uncertainties of U_{AV} measurements when the RL of ILC are NMIs, accredited by CLs and accredited RLs that are not NMIs or accredited by CLs, is as follows:

$$U_{AV\ NMI} < U_{AV\ CL} < U_{AV\ RL}, \tag{4}$$

that is, the most accurate ILC are those that are performed by NMIs.

The value of the extended uncertainty of the $U_{AV\ NMI}$ measurements for a case where the NMI is RL can be derived from the results of the corresponding international comparisons of national measurement standards in which the NMI participated.

The value of the extended uncertainty of the $U_{AV\ CL}$ measurement for a case where CL is RL can be derived from the corresponding calibration certificates for MIs issued by the NMI using CL in the ILC.

The value of the expanded uncertainty of $U_{AV\ RL}$ measurements for a case where an RL is an accredited provider can be obtained from the corresponding calibration certificates for the MI issued by accredited CLs that use RLs in the ILC.

In [38] proposed procedure links results from international comparisons of national measuring standards and results from ILC for CL. This procedure can be used for practical estimation of results specific ILCs on a national level in different countries by means of the results from the laboratories of NMIs as well as overcoming technical barriers between countries.

According to [37] on the basis of measurement results and associated uncertainties presented by participants of RMO comparisons of national measuring standards, calculate the value of the χ^2 criterion.

The same formula from [37] can be applied to evaluation of consistency of the results of ILCs for CLs:

$$\chi^2 = \sum_{i=1}^{n} \frac{D_{lab\ i}^{\ 2}}{u^2(x_{lab\ i})}. \tag{5}$$

To checks consistency of ILCs for CLs were used criterion value, calculated from data provided by the CLs does not exceed the critical value χ^2 for confidence level 0.95 and the number of degrees of freedom n – 1 (n is number of CL-participants of ILC):

$$\chi^2 \prec \chi^2_{0.95}(n-1). \tag{6}$$

CLs that provides maximum E_n-criterion is determined

$$\max_i E_n = \frac{|D_{lab\ i}|}{2\sqrt{u_{lab\ i}^2 + u_{AV}^2}}. \tag{7}$$

Further that CL's data is temporary excluded from the consideration, and the procedure evaluation of χ^2

criterion is repeated. The sequential exclusion of data is repeated until the condition (6) is fulfilled.

Those CLs, whose results were excluded, have to analyze the reasons of their results falling out.

Conclusion

The choice of methodology for processing the ILC results is made. A universal algorithm for processing the ILC data obtained is proposed, which allows the reference laboratory to take into account all the reporting peculiarities during ILC and to make a selection of the methodology of processing the data of ILC.

To check the consistency of the results of interlaboratory comparisons using the χ^2 criterion is proposed. In the case of receiving inconsistency of data they reject or specifying the applied indicators for further consistency. This will lead to more credible ILC of accredited TLs and CLs, and will increase the competitiveness of national industrial producers.

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ОСОБЛИВОСТІ ОБРОБЛЕННЯ РЕЗУЛЬТАТІВ І ОЦІНЮВАННЯ НЕВИЗНАЧЕНОСТІ ВИМІРЮВАНЬ МІЖЛАБОРАТОРНИХ ПОРІВНЯЛЬНИХ ВИПРОБУВАНЬ ДЛЯ КАЛІБРУВАЛЬНИХ ЛАБОРАТОРІЙ

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Міжлабораторні порівняння є однією з форм експериментальної перевірки діяльності лабораторій з метою визначення технічної компетентності у певному виді діяльності. Програму Міжлабораторних порівнянь розробляють з урахуванням вимог національних стандартів ДСТУ ISO/IEC 17025, ДСТУ EN ISO/IEC 17043, ДСТУ ISO 13528, які гармонізовані з відповідними міжнародними та європейськими стандартами. Міжлабораторні порівняння для калібрувальних лабораторій (КЛ) здійснюються компетентними провайдерами. Для оцінювання результатів участі КЛ в таких випробуваннях використовуютья критерії, встановлені провайдерами порівнянь для забезпечення якості калібрування для певного виду вимірювань. Основним завданням провайдера є визначення опорного вимірюваного значення та його невизначеності. Існують різні алгоритми оброблення результатів міжлабораторних порівнянь, які базуються на статистичних методах. Вибір конкретного методу оцінювання результатів випробувань залежить від виду досліджуваного зразка, особливостей випробувань і кількості лабораторій, що беруть участь в випробуваннях. Міжлабораторні порівняння КЛ можуть базуватися на трьох основних типах організації порівнянь, коли провайдерами (референтними лабораторіями) можуть бути: національні метрологічні інститути (НМІ); акредитована КЛ; акредитовані провайдерами, які не є НМІ або акредитованими КЛ. Пропонується проводити перевірку узгодженості отриманих результатів міжлабораторних порівнянь за допомогою критерію χ^2. *У разі отримання неузгоджених даних проводять аналіз з метою відхилення цих даних або для подальшого узгодження шляхом уточнення застосованих показників. Це сприятиме отриманню більш надійних результатів міжлабораторних порівнянь акредитованих КЛ та випробувальних лабораторій, а також підвищить конкурентоспроможність національних виробників.*

Ключові слова: *міжлабораторні порівняння, калібрувальна лабораторія, невизначеність вимірювання, зразок порівняння, національний метрологічний інститут.*

ОСОБЕННОСТИ ОБРАБОТКИ РЕЗУЛЬТАТОВ И ОЦЕНИВАНИЯ НЕОПРЕДЕЛЕННОСТИ ИЗМЕРЕНИЙ МЕЖЛАБОРАТОРНЫХ СРАВНИТЕЛЬНЫХ ИСПЫТАНИЙ ДЛЯ КАЛИБРОВОЧНЫХ ЛАБОРАТОРИЙ

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Межлабораторные сравнения для калибровочных лабораторий (КЛ) осуществляются компетентными провайдерами. Для оценки результатов участия КЛ в таких испытаниях используются критерии, установленные провайдерами сравнений для обеспечения качества калибровки для определенного вида измерений. Основной задачей провайдера является определение опорного измеряемого значения и его неопределенности. Межлабораторные сравнения КЛ могут базироваться на трех основных типах организации сравнений, когда провайдерами (референтными лабораториями) могут быть: национальные метрологические институты (НМИ); аккредитованная КЛ; аккредитованные провайдеры, которые не являются НМИ или аккредитованными КЛ. Предложено осуществлять проверку согласованности полученных результатов межлабораторных сравнений с помощью критерия χ^2.

Ключевые слова: *межлабораторные сравнения, калибровочная лаборатория, неопределенность измерения, образец сравнения, национальный метрологический институт.*