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Main stages of calibration of measuring instruments

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Abstract

The main stages of calibration of measuring instruments are described. The stage of preparation for calibration and its main steps are considered: setting a measurement task, choosing a method and equipment, choosing (developing) calibration methods and their verification (validation). The content of the measurement experiment is presented together with the main measurement methods that can be used to calibrate the indications of measuring instruments and material measures. The main steps of experimental data processing, which lead to the estimation of the numerical value and uncertainty evaluation of the measurand being calibrated, are considered. The preparation of calibration results, including the uncertainty budget and calibration certificate, is described. Procedures for assessing the probability of compliance of a calibrated measuring instrument and material measure with the specified metrological characteristics, as well as for validating their calibration methods, are considered.

Keywords: calibration; methods and models; measurement uncertainty; verification; validation; conformance probability.

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Introduction

Metrological traceability is an integral condition for ensuring the uniformity of measurement results, which is a property of a measurement result whereby the result can be related to a stated metrological reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty [1], cl. 2.43.

In accordance with cl. 2.39 [1], calibration is an operation that, under specified conditions, in a first step establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication.

Many regulatory documents and publications are dedicated to certain issues of calibration, which require a lot of time and effort to understand, especially when there is a lack of practical experience. Therefore, we consider it appropriate to briefly outline the main stages of calibration and their features in one paper.

Calibration includes five main stages:

- preparation for calibration;
- measurement experiment;
- processing of experimental data;
- preparation of calibration results;
- obtaining calibration results in practice.

At all stages of calibration, there are several features that shall be systematized for its correct implementation.

Calibration of measuring instruments (MIs) is carried out by enterprises, organizations or their separate divisions, which are called calibration laboratories (CL) [2].

At the same time, in accordance with cl. 7.6.2 of ISO/IEC 17025:2017 [3], CL shall evaluate measurement uncertainty (MU) for all types of measurements.

The value of the expanded uncertainty U obtained during the calibration of MIs is used further for:

- MU evaluation carried out by the Customer using a calibrated MI;
- making decision (by the Customer or CL) regarding the compliance of the calibrated MI with the specified metrological requirements;
- validation and verification of the calibration method by CL.

In all of these cases, the result obtained depends on the reliability of determining the expanded uncertainty of the calibrated MI. At the same time, a reliable evaluation of the expanded uncertainty shall account for the assigned distribution laws for input quantities [4].

This paper addresses all the above-mentioned issues.

1. Preparation for calibration

Preparation for calibration includes the following steps.

1.1. Setting the measurement task (by the Customer):

• provision of a specific type of the MI being calibrated;

• setting calibration points within the range of values of the measurand [$X_{c\min}$; $X_{c\max}$];

• setting calibration conditions: temperature T_c, pressure P_c, humidity W_c;

• setting the maximum permissible (target) uncertainty, $U_{c\max}$.

1.2. Selection of method and equipment

CL equipment includes MIs, software, measurement standards, reference materials, reference data, reagents, consumables or auxiliary devices that may affect the calibration results.

The entire variety of MIs for calibration can be divided into three groups:

• material measures (MMs) are the MIs that reproduce values during the use or permanently retain assigned values [1], cl. 3.6;

• indicating measuring instruments (IMIs) are the MIs that provide an output signal carrying information about the values of the measurand [1], cl. 3.3;

• transfer device is technical equipment or a certain environment, with the help of which it is possible either to compare measures of the quantities of the same kind, or the indications of IMIs with each other [5], cl. 6.15.

Depending on the metrological subordination, MMs and IMIs at calibration are divided into reference and calibrated ones.

The reference MIs shall meet the following requirements:

• the range of reproducible (measured) values [$X_{s\min}$; $X_{s\max}$], moreover, $X_{s\min} \leqslant X_{c\min}$; $X_{s\max} \geqslant X_{c\max}$;

• the possibility to reproduce (measure) a value under given calibration conditions: (T_c, P_c, W_c) with a given uncertainty $U_{s\max}$;

• the largest achievable instrumental uncertainty shall meet the following condition: $U_{s\max} = (0.2...0.33)MPE_c$, where MPE_c is the maximum permissible error of the MMs being calibrated [6], formula (5.2).

Auxiliary devices shall provide all the necessary conditions for calibration (T_c, P_c, W_c). Equipment requirements are listed in cl. 6.4 [3].

The calibration method is selected depending on the types of reference MIs and MIs for calibration (it can be suggested by the Customer or CL). Measurement Method is a generic description of a logical organization of the operations used in the process of measurement [1], cl. 2.5.

In [1, 7], the following measurement methods that can be used for calibration are given:

• method of direct measurements ([7], 311-02-01) that is a measurement method, in which the value of a quantity is determined directly from the indicating measuring instrument;

• measurement method by comparison/method of comparison with a measure ([7], 311-02-03) that is a measurement method based on comparison of the measurand with a known quantity of the same kind;

• method of indirect measurements ([7], 311-02-02) that is a measurement method, in which the value of a quantity is directly obtained by measuring other quantities related to the measurand through a known relation;

• substitution measurement method ([7], 311-02-04) that is a comparison measurement method, in which the measurand is replaced by a known quantity of the same kind, selected so that the effects of these two quantities on the measuring instrument are the same;

• supplement method measurement ([7], 311-02-05) that is a measurement method by comparison, in which the measurand is supplemented by a known value of a quantity of the same kind, selected so that the sum of their values would be equal to a given value;

• differential measurement method ([7], 311-02-06) that is a comparison measurement method based on the measurement of the algebraic difference between the values of the measurand and a quantity of the same kind with a known value that slightly differs from the value of the measurand;

• null measurement method ([7], 311-02-07) that is a differential measurement method, in which the difference between the value of the measurand and the known value of the quantity of the same kind, with which it is compared, is reduced to zero.

1.3. Selection (development) of a calibration method

The laboratory shall use calibration methods that satisfy the Customer's needs and are suitable for the calibration.

If the Customer does not specify the method to be used, the laboratory then selects appropriate methods that are set forth in international, regional or national standards, recommended by authoritative technical organizations, have been described in appropriate scientific papers or journals, or have been recommended by the manufacturer.

In cl. 5.4.4 of ISO/IEC 17025:2005 [8], it is recommended that the calibration method include at least the following information:

• appropriate identification;

• scope;

• description of the MI type to be tested or calibrated;

• parameters or quantities and ranges to be determined;

• hardware and equipment, including technical performance requirements;

• reference measurement standards and reference materials required;

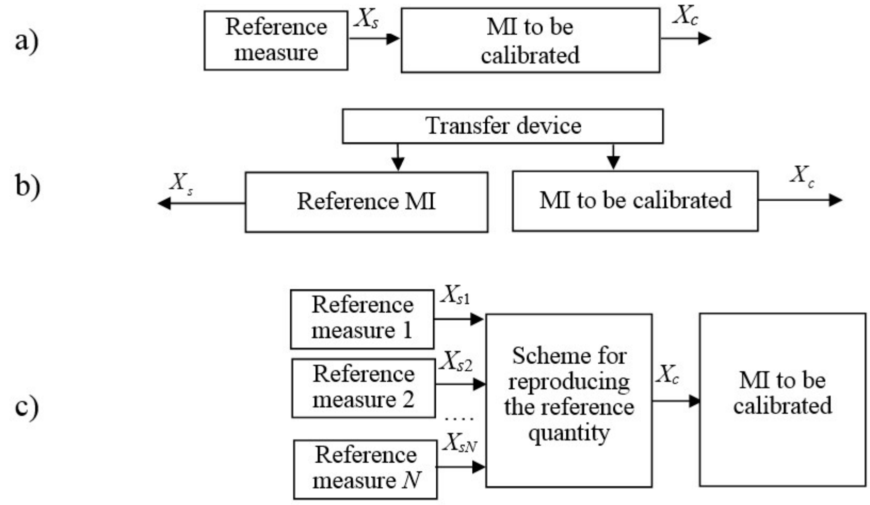

Fig. 1. MI calibration schemes: a) direct measurement of a quantity reproduced by a reference measure using a MI being calibrated; b) comparison of the reference MI and the MI being calibrated using a transfer device; c) indirect reproduction of the quantity measured by the MM being calibrated using reference MMs

- environmental conditions required and any stabilization period;
- description of the procedure;
- criteria and/or requirements for approval/ rejection;
- data to be recorded and method of analysis and presentation;
- uncertainty or the procedure for its evaluation.

1.4. Verification (validation) of the calibration method

The laboratory ensures that the standardized methods are correctly used before calibrations. This confirmation procedure is called *verification*.

In the case of using methods established (developed) by the laboratory and standardized methods used outside their target scope, as well as using the extensions and modifications of the standardized methods, CL shall further ensure that these methods are suitable for the intended use. This confirmation procedure is called *validation*.

To determine the effectiveness of the method, one or several techniques in combination shall be used [3], cl. 7.2.2.1:

a) calibration or estimation of the bias and precision using reference measurement standards or reference materials;

b) systematic assessment of the factors affecting the result;

c) testing the robustness of the method through the variation of the controlled parameters, such as incubator temperature or volume dispensed;

d) comparison of the results with other validated methods;

e) interlaboratory comparisons;

f) evaluation of MU of the measurand based on an understanding of theoretical principles of the method and practical experience of the sampling or the test method application.

Verification (validation) of calibration methods shall be carried out under the conditions specified by the Customer (T_c, P_c, W_c), and for the entire range of values of the measurand [$X_{c\min}$; $X_{c\max}$].

2. Measurement experiment

The measurement experiment is carried out in accordance with the chosen measurement method. The same measurement methods used both for verification and calibration of MMs and MIs are described in detail in [9].

When calibrating MIs, three most common methods imply using the calibration schemes shown in Fig. 1.

Examples of measurements according to the given schemes:

a) direct measurement of a quantity, L_s, reproduced by a reference gauge block using a micrometer being calibrated, L_c;

b) comparison of the indications of the thermometer being calibrated T_c using a reference thermometer T_s, with a thermostat being used as a transfer device;

c) measurement of the reference power W_s, which is reproduced by a voltage calibrator V_s and a reference resistor R_s according to the formula $W_s = V_s^2 / R_s$, using a wattmeter being calibrated, W_c.

When calibrating MMs, three most common methods imply using the calibration schemes shown in Fig. 2.

Examples of measurements according to the given schemes:

a) calibration of a multivalued measure of resistance, R_c, using a reference ohmmeter, R_s;

b) comparing the mass of the reference weight m_s with the mass of the weight being calibrated m_c using the mass comparator balance;

c) calibration of a measure of electrical resistivity of a conductor ρ_c by measuring its resistance R_s using a reference ohmmeter, diameter D_s using a reference micrometer and length l_s using a standard dial caliper according to the formula $\rho_c = R_s \pi D_s^2 / (4 l_s)$.

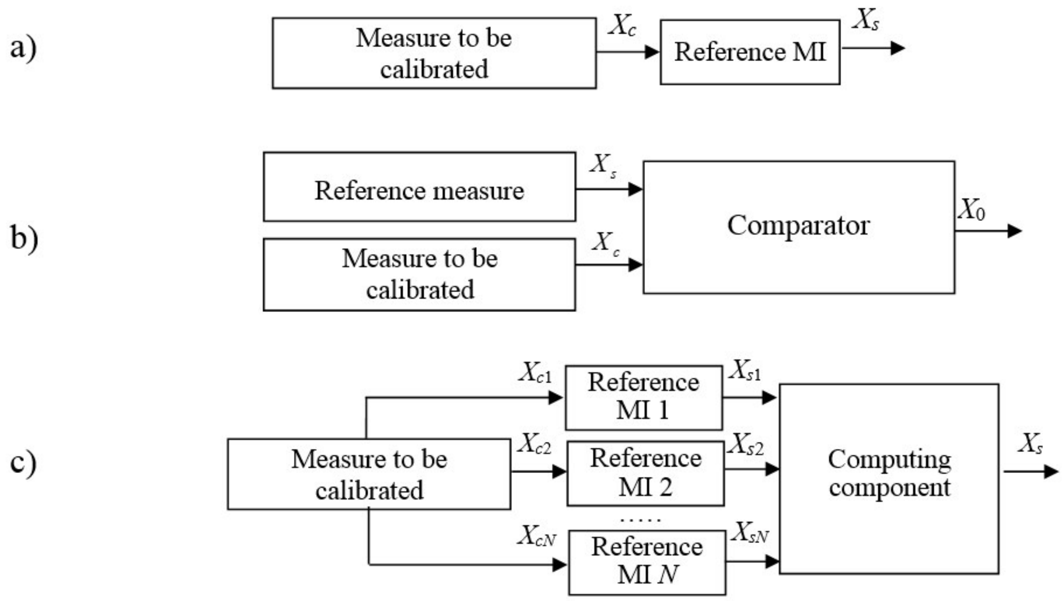

Fig. 2. MM calibration schemes: a) direct measurement of a quantity reproduced by the MM being calibrated using a reference MI; b) comparison of the reference MM and the MM being calibrated using a comparator; c) indirect measurement of a quantity value reproduced by the MM being calibrated using reference MIs

3. Experimental data processing

Processing of experimental data obtained as a result of a measurement experiment during the calibration includes the following operations [10, 11].

1. Recording the mathematical calibration model. For the calibration schemes shown in Fig. 1 and 2, mathematical models are considered in [9].

2. Estimation of the input quantities. The values of the input quantities are found by measuring them after either single or multiple observations, or taken from the external sources.

3. Calculation of an estimate of the measurand. An estimate of the measurand is obtained by substituting estimates of the input values into the mathematical calibration model.

4. Evaluation of standard uncertainties of the input quantities by Type *A* or Type *B* evaluation method. In method *A*, the standard uncertainty of measurement is evaluated using statistical analysis of a series of observations. In this case, the standard measurement uncertainty is the experimental standard deviation of the mean obtained using averaging methods. For method *B*, standard uncertainty is evaluated in ways other than statistical analysis of a series of observations and is based on another scientific knowledge.

5. Calculation of the uncertainty contribution of the input quantity to the uncertainty of the measurand. Defined as the product of the uncertainty of the input quantity by the sensitivity coefficient, showing how the estimate of the measurand will change with a change in the estimate of the input quantity, and is found as a partial derivative of the output quantity against the corresponding input quantity.

6. Evaluation of the standard MU of the measurand (combined standard uncertainty). The calculation is carried out according to the distribution law for the uncertainty. In the absence of correlations between input quantities, the standard uncertainty of the input quantity is defined as the square root of the sum of the squares of all uncertainty contributions. If there are correlations between input quantities, all their covariances are added under the root.

7. Evaluation of the expanded uncertainty. The uncertainty is obtained by multiplying the uncertainty of the measurand (combined standard uncertainty) by the coverage factor, its approximate value for a confidence level of 0.9545 being equal to 2.

In the presence of uncertainty contributions of Type *A*, GUM [10] recommends taking the Student coefficient as the coverage factor for a confidence level of 0.9545 and an effective number of degrees of freedom determined by the Welch-Satterthwaite formula.

A reliable evaluation of the expanded uncertainty cannot be obtained without accounting for the laws of distribution for input quantities, which is usually done by the Monte Carlo method (MCM) [4]. For calibration issues, a reliable evaluation of the expanded uncertainty can be obtained using the kurtosis method [12]. Its use makes it possible to automate the calculation of uncertainty, and the expanded uncertainty itself will be close to that obtained by MCM.

4. Registration of calibration results

For the registration of the calibration results, the following operations are performed:

- preparing the budget of measurement uncertainty;
- registration of a calibration certificate.

The measurement uncertainty budget first appeared in [11] as an expected result of measurement uncertainty evaluation. It is convenient to represent the intermediate results obtained in the process of implementing the basic algorithm for measurement uncertainty evaluation in the form of an uncertainty budget, which includes a list of all input quantities, their estimates, along with the standard measurement uncertainties assigned to them, sensitivity coefficients and numbers of degrees of freedom (or kurtosis). In addition to information about the input quantities, it is convenient to include information about the measurand in the budget: the measurement result, the combined standard uncertainty, the effective number of degrees of freedom (kurtosis), the coverage factor and the expanded uncertainty.

Since the uncertainty budget is prepared in the form of a table, it allows, when using Excel, to automate the routine process of uncertainty evaluation, reducing the time of calculations and increasing their reliability.

General requirements for reports of tests, calibration or sampling are listed in [3], cl. 7.8.2. Special requirements for calibration certificates are given in [3], cl. 7.8.4, among which the following information is specified:

a) the MU of the measurand presented in the same unit as that of the measurand or in a form relative to the measurand (e.g. percent);

b) the conditions (e.g. environmental), under which the calibrations were performed, that affect the measurement results;

c) a statement identifying how the measurements are metrologically traceable;

d) the results before and after any adjustment or repair, if available;

e) where applicable, a statement of conformity with requirements or specifications (see 7.8.6 [3]);

f) where applicable, opinions and interpretations (see 7.8.7 [3]).

A calibration certificate or calibration label shall not contain any recommendation on the calibration interval, except this has been agreed with the Customer.

5. Using calibration results in practice

Calibration results are most often used in practice when the Customer evaluates MU by using a calibrated MI. This issue is well addressed in existing documents on measurement uncertainty [10, 11].

In addition, the calibration results are used by CL for:

• making decision regarding the compliance of the calibrated MI with the specified metrological requirements;

• validation and verification of the calibration method.

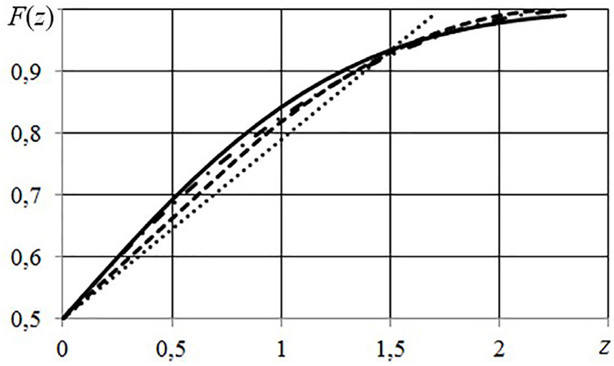

Fig. 3. Dependencies for uniform (···), trapezoidal (---) (γ =0.5), triangular (−·−) and normal (—) distribution laws

5.1. Assessing the suitability of a calibrated MI

In cl. 7.8.4 of the standard [3], it is stated that the calibration certificate may, where appropriate, include a statement of compliance with the requirements or specifications. Such a statement is a statement that the MI is suitable to be used with a probability of compliance p_c.

To assess the probability of compliance p_c of metrological characteristics of the MI with the documentation requirements, the following expressions are used [6, 13]:

$$p_c = F\left(\frac{MPE - \left|\hat{\Delta}\right|}{u}\right),$$

when assessing the conformity of the MI, and:

$$p_c = F\left(\frac{\hat{X}_N - \hat{X}_c}{u}\right),$$

when assessing the conformity of the MM.

In these expressions: MPE is the maximum permissible error of the MI; $\hat{\Delta}$ is the estimate of the systematic error (shift) at the MI calibration point; $\hat{X}_N$ is the nominal value of the MM; $\hat{X}_c$ is the MM value found as a result of the calibration; F and u are the distribution law and the standard uncertainty of the measurand being calibrated, respectively.

Fig. 3 shows the dependences obtained in [14] for the uniform, triangular, trapezoidal and normal laws of distribution of the measurand.

The owner of the MI compares the obtained probability value of compliance p_c with the acceptable probability value p_0. If $p_c \leqslant p_0$, the MI is considered suitable for the intended use, if $p_c < p_0$, the MI is considered unsuitable for the intended use.

5.2. Validation (verification) of calibration methods

The laboratory shall use acceptable methods and procedures for all laboratory activities and, where appropriate, for the MU evaluation, as well as statistical methods for data analysis [3], cl. 7.2.1.1.

Before their use, the laboratory shall ensure that the methods are implemented correctly (method verification) by providing evidence that the expected results can be achieved. Verification records shall be kept [3], cl. 7.2.1.5.

The laboratory shall validate non-standard methods, laboratory-developed methods and standard methods used outside their intended scope or otherwise modified [3], cl. 7.2.2.1.

The procedures used to validate (verify) methods may be one or a combination of the following:

a) calibration or evaluation of the bias and precision using reference measurement standards or reference materials;

b) systematic assessment of the factors affecting the result;

c) testing the robustness of the method through variation of controlled parameters, such as incubator temperature, volume dispensed;

d) comparison of the results with other validated methods;

e) interlaboratory comparisons;

f) evaluation of MU of the measurand based on an understanding of theoretical principles of the method and practical experience of the sampling or the test method application.

Depending on the chosen validation method, the required validation characteristics are selected. Typical characteristics for method validation of analytical methods are: precision, bias, linearity, detection threshold, robustness, sampling [15]. In other documents, this list is slightly different, and all note that the choice of the required set of characteristics from those listed depends on the specific method being validated. When validating calibration methods, it is advisable to select metrological characteristics of the MI, which are specified in its calibration certificate, as validation characteristics.

When validating (verifying) the methods for calibrating IMIs, the previously calibrated IMI is recalibrated according to the laboratory methods with the bias values, $\hat{\Delta}_{cert}$, and the expanded uncertainty, U_{cert}, specified in its calibration certificate [9]. To establish the suitability of the method, the formula shall be used:

$$E_n = \frac{\left|\hat{\Delta}_{lab} - \hat{\Delta}_{cert}\right|}{\sqrt{U_{lab}^2 + U_{cert}^2}},$$

in which $\hat{\Delta}_{lab}$ and U_{lab} are the bias of the reference IMI and its expanded uncertainty respectively, which were obtained as a result of applying the calibration method to the reference IMI in CL.

When validating (verifying) calibration methods for MMs, a previously calibrated MM with the real values of the measure $\hat{X}_{cert}$ and expanded uncertainty U_{cert} specified in its calibration certificate is recalibrated according to laboratory methods [9]. To establish the suitability of the method, the following formula shall be used:

$$E_n = \frac{\left|\hat{X}_{lab} - \hat{X}_{cert}\right|}{\sqrt{U_{lab}^2 + U_{cert}^2}},$$

in which $\hat{X}_{lab}$ and U_{lab}, are the value of the reference MM and its expanded uncertainty respectively, which were obtained as a result of applying the calibration method to the reference MM in CL.

If $E_n \leqslant 1$ over the entire range of changes in the value of X_s, within a given range of changes in influence quantities and specified environmental parameters, the method is suitable for the intended use.

The results of the conformity assessment, the procedure used for validation and the decision about the suitability (or unsuitability) of the method applied for the intended use shall be documented in the form of a validation (verification) protocol.

The validation protocol shall contain the following information [3]: a) the validation procedure used; b) specification of the requirements; c) determination of the performance characteristics of the method; d) results obtained; e) a statement on the validity of the method, detailing its suitability for the intended use.

Основні етапи калібрування засобів вимірювання

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Анотація

Описано основні етапи калібрування засобів вимірювання (ЗВ): підготовка до калібрування; вимірювальний експеримент; обробка експериментальних даних; оформлення результатів калібрування; використання результатів калібрування на практиці. Розглянуто етап підготовки до калібрування та його основні етапи: формулювання завдання вимірювання, вибір методу та обладнання, обрання (розробку) методу калібрування, верифікацію (валідацію) методів калібрування. Подано зміст вимірювального експерименту з наведенням основних методів вимірювання, які можна використовувати при калібруванні засобів вимірювання та матеріальних мір: пряме вимірювання, звірення еталонного

ЗВ та ЗВ, що калібрується, за допомогою пристрою порівняння; непряме вимірювання. Розглянуто основні етапи обробки експериментальних даних, що призводять до оцінювання числового значення та невизначеності вимірюваної величини під час калібрування: запис математичної моделі калібрування, оцінювання вхідних величин, обчислення числового значення вимірюваної величини, оцінювання стандартних невизначеностей вхідних величин, обчислення внеску невизначеності вхідної величини в невизначеність вимірюваної величини, обчислення стандартної та розширеної невизначеності вимірюваної величини. Описано оформлення підсумків калібрування у вигляді побудови бюджету невизначеності вимірювань та оформлення сертифіката калібрування. Обговорено використання результатів калібрування на практиці: оцінювання ймовірності відповідності відкаліброваного засобу вимірювання та матеріальної міри заданим метрологічним характеристикам, а також затвердження валідації методів калібрування для визначення їхньої ефективності.

Ключові слова: калібрування; методи та моделі; метод калібрування; невизначеність вимірювання; бюджет невизначеності; верифікація; валідація; ймовірність відповідності.

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