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Točnost (pravilnost in natančnost) merilnih metod in rezultatov - 2. del: Temeljna metoda določevanja ponovljivosti in obnovljivosti standardne merilne metode

Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

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Exactitude (justesse et fidélité) des résultats et méthodes de mesure - Partie 2: Méthode de base pour la détermination de la répétabilité et de la reproductibilité d'une méthode de mesure normalisée

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**Accuracy (trueness and precision) of
measurement methods and results —**

Part 2:

**Basic method for the determination of
repeatability and reproducibility of a
standard measurement method**

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*Exactitude (justesse et fidélité) des résultats et méthodes de mesure —
Partie 2: Méthode de base pour la détermination de la répétabilité et
de la reproductibilité d'une méthode de mesure normalisée*

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Contents

Page

Foreword.....	v
Introduction.....	vi
1 Scope.....	1
2 Normative references.....	2
3 Terms and definitions.....	2
4 Symbols.....	2
5 Estimates of the parameters in the basic model.....	4
6 Requirements for a precision experiment.....	5
6.1 Layout of the experiment.....	5
6.2 Recruitment of the laboratories.....	6
6.3 Preparation of the materials.....	6
7 Personnel involved in a precision experiment.....	7
7.1 Panel.....	7
7.2 Statistical functions.....	8
7.3 Executive functions.....	8
7.4 Supervisors.....	9
7.5 Operators.....	10
8 Statistical analysis of a precision experiment.....	10
8.1 Preliminary considerations.....	10
8.2 Tabulation of the results and notation used.....	11
8.2.1 Cells.....	11
8.2.2 Redundant data.....	11
8.2.3 Missing data.....	11
8.2.4 Outliers.....	11
8.2.5 Outlying laboratories.....	11
8.2.6 Erroneous data.....	11
8.2.7 Balanced uniform-level test results.....	11
8.2.8 Collation of data and intermediate values.....	12
8.2.9 Original test results.....	12
8.2.10 Cell means (Form B of Figure 2).....	12
8.2.11 Measures of cell spread (Form C of Figure 2).....	12
8.2.12 Corrected or rejected data.....	13
8.3 Scrutiny of results for consistency and outliers.....	13
8.3.1 Approaches for scrutiny of data.....	13
8.3.2 Graphical consistency technique.....	13
8.3.3 Numerical outlier technique.....	16
8.3.4 Cochran's test.....	16
8.3.5 Grubbs' tests.....	18
8.3.6 Repeated testing for outlying means or outlying data points.....	20
8.3.7 Alternative outlier inspection and test methods.....	20
8.4 Calculation of the general mean and variances.....	20
8.4.1 Method of analysis.....	20
8.4.2 Basic data.....	21
8.4.3 Non-empty cells.....	21
8.4.4 Calculation of the general mean, \hat{m}	21
8.4.5 Calculation of variances.....	21
8.4.6 Alternative calculation methods for variances.....	22
8.4.7 Dependence of the variances upon m	23
8.5 Establishing a functional relationship between precision values, s , and the mean level, m	23
8.5.1 Choice of functional relationship.....	23

ISO 5725-2:2019(E)

8.5.2	Fitting relationships I and II	24
8.5.3	Fitting relationship III	25
8.5.4	Fitting relationship IV	26
8.6	Statistical analysis as a step-by-step procedure	28
8.7	Report to the panel and decisions to be taken by the panel	30
8.7.1	Report by the statistical expert	30
8.7.2	Decisions to be taken by the panel	32
8.7.3	Full report	33
9	Statistical tables	33
Annex A (informative)	Number of laboratories required for an estimate of precision	38
Annex B (informative)	Alternative calculations of variance components	41
Annex C (informative)	Examples of the statistical analysis of precision experiments	44
Annex D (informative)	Calculation of critical values and indicators	66
Bibliography		69

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 69, *Applications of statistical methods*, Subcommittee SC 6, *Measurement methods and results*.

This second edition cancels and replaces the first edition (ISO 5725-2:1994), which has been technically revised. It also incorporates the Technical Corrigendum ISO 5725-2:1994/Cor 1:2002.

The main changes compared to the previous edition are as follows:

- permission is given to use alternative scrutiny and outlier detection tests provided that the performance is similar;
- permission is given to apply modern statistical methods available for calculations of the relevant precision and trueness characteristics;
- guidance on the number of laboratories required for a precision study has been included;
- information on the computation of critical values has been included.

A list of all parts in the ISO 5725 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

ISO 5725-2:2019(E)

Introduction

ISO 5725 uses two terms, “trueness” and “precision”, to describe the accuracy of a measurement method. “Trueness” refers to the closeness of agreement between the arithmetic mean of a large number of test results and the true or accepted reference value. “Precision” refers to the closeness of agreement between test results.

General consideration of these quantities is given in ISO 5725-1 and so is not repeated in this document. ISO 5725-1 should be read in conjunction with all other parts of ISO 5725, including this part, because it gives the underlying definitions and general principles.

This document is concerned solely with estimating the repeatability standard deviation and reproducibility standard deviation based on an interlaboratory design in which each laboratory conducts a number of independent measurements of the same sample under repeatability conditions. There are other designs (such as nested, factorial or split-level experiments) which can be used for the estimation of precision: these are not dealt with in this document but rather are the subject of other parts of ISO 5725. Nor does this document consider any other measures of precision intermediate between the two principal measures; those are the subject of ISO 5725-3.

In certain circumstances, the data obtained from an experiment carried out to estimate precision are used also to estimate trueness and can be used to evaluate measurement uncertainty. The estimation of trueness is not considered in this document; all aspects of the estimation of trueness are the subject of ISO 5725-4. The evaluation of measurement uncertainty, using inter-laboratory estimates of trueness and precision, is the subject of ISO 21748.

[Annex C](#) provides practical examples of estimating the precision of measurement methods by experiment. Worked examples are given to demonstrate balanced uniform sets of test results, although in one example a variable number of replicates per cell were reported (unbalanced design) and in another some data were missing. This is because an experiment designed to be balanced can turn out to be unbalanced. Stragglers and outliers are also considered.

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Accuracy (trueness and precision) of measurement methods and results —

Part 2:

Basic method for the determination of repeatability and reproducibility of a standard measurement method

1 Scope

1.1 This document

- amplifies the general principles for designing experiments for the numerical estimation of the precision of measurement methods by means of a collaborative interlaboratory experiment;
- provides a detailed practical description of the basic method for routine use in estimating the precision of measurement methods;
- provides guidance to all personnel concerned with designing, performing or analysing the results of the tests for estimating precision.

NOTE Modifications to this basic method for particular purposes are given in other parts of ISO 5725.

1.2 It is concerned exclusively with measurement methods which yield measurements on a continuous scale and give a single value as the test result, although this single value can be the outcome of a calculation from a set of observations.

1.3 It assumes that in the design and performance of the precision experiment, all the principles as laid down in ISO 5725-1 are observed. The basic method uses the same number of test results in each laboratory, with each laboratory analysing the same levels of test sample; i.e. a balanced uniform-level experiment. The basic method applies to procedures that have been standardized and are in regular use in a number of laboratories.

1.4 The statistical model of ISO 5725-1:1994, Clause 5, is accepted as a suitable basis for the interpretation and analysis of the test results, the distribution of which is approximately normal.

1.5 The basic method, as described in this document, (usually) estimates the precision of a measurement method:

- a) when it is required to determine the repeatability and reproducibility standard deviations as defined in ISO 5725-1;
- b) when the materials to be used are homogeneous, or when the effects of heterogeneity can be included in the precision values; and
- c) when the use of a balanced uniform-level layout is acceptable.

1.6 The same approach can be used to make a preliminary estimate of precision for measurement methods which have not reached standardization or are not in routine use.

ISO 5725-2:2019(E)

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3534-1, *Statistics — Vocabulary and symbols — Part 1: Probability and general statistical terms*

ISO 3534-2, *Statistics — Vocabulary and symbols — Part 2: Applied statistics*

ISO 3534-3, *Statistics — Vocabulary and symbols — Part 3: Design of experiments*

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

3 Terms and definitions

For the purposes of this document, the definitions given in ISO 3534-1, ISO 3534-2, ISO 3534-3, and ISO 5725-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <https://www.iso.org/obp>

— IEC Electropedia: available at <http://www.electropedia.org/>

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4 Symbols

α	Probability associated with a critical value of a test statistic, also referred to as a level of significance
a	Intercept in the relationship $s = a + bm$
a_v	Intercept parameter in the relationship $s_j^2 = a_v^2 + (b_v m)^2$
A	Factor used to calculate the uncertainty of an estimate
b	Slope in the relationship $s = a + bm$
b_v	Slope parameter in the relationship $s_j^2 = a_v^2 + (b_v m)^2$
B	Laboratory component of bias under repeatability conditions
c	Intercept in the relationship $\lg s = c + d \lg m$
C, C', C''	Test statistics
$C_{\text{crit}}, C'_{\text{crit}}, C''_{\text{crit}}$	Critical values for statistical tests
d	Slope in the relationship $\lg s = c + d \lg m$
e	Component in a test result representing the random error occurring in every test result
G	Grubbs' test statistic
h	Mandel's between-laboratory consistency test statistic
k	Mandel's within-laboratory consistency test statistic

$L(\theta)$	Log-likelihood for variance components θ
m	General mean of the test property; level
\hat{m}	Estimate of the general mean of the test property
\mathbf{M}	Transformation matrix used in REML estimation
N	Number of iterations
n	Number of test results obtained in one laboratory at one level (i.e. per cell)
n_j	Total number of test results obtained at level j of the interlaboratory experiment
p	Number of laboratories participating in the interlaboratory experiment
P	Probability
q	Number of levels of the test property in the interlaboratory experiment
r	Repeatability limit
R	Reproducibility limit
s	Estimate of a standard deviation
\hat{s}	Predicted standard deviation
T	Total or sum of some expression
t	Number of test objects or groups
$\mathbf{V}(\theta)$	Covariance matrix used in REML estimation
W	Weighting factor used in calculating a weighted regression
w	Weighting factor used in calculating a weighted mean
x	Datum used for Grubbs' test
\mathbf{X}	Design matrix for REML estimations
y	Test result
$\bar{\bar{y}}$	Grand mean of test results
\mathbf{Y}	Vector of all observations at a level j
θ	Vector of variance components used in REML estimation
μ	True value or accepted reference value of a test property
σ	True value of a standard deviation

ISO 5725-2:2019(E)

Subscripts

i	Identifier for a particular laboratory Index for summation (Annex A)
j	Identifier for a particular level Index for summation (Annex A)
k	Identifier for a particular test result in a laboratory i at level j
L	Between-laboratory (interlaboratory)
P	Probability
r	Repeatability
R	Reproducibility
REML	Estimate arising from a restricted maximum likelihood calculation
v	Terms used in calculation of a relationship between mean and combined variance (see 8.5.1.3 , relationship III)
W	Within-laboratory (intralaboratory)
1, 2, 3, ...	For test results, numbering in the order of obtaining them; for other cases (laboratories), as arbitrary identifiers
(1), (2), (3), ...	For test results, (1), (2) ... denote the 1 st , 2 nd ... etc. order statistic, that is, the 1 st , 2 nd ... etc. value numbered in the order of increasing magnitude

5 Estimates of the parameters in the basic model

5.1 The procedures given in this document are based on the statistical model given in Clause 5 of ISO 5725-1:1994 and elaborated upon in ISO 5725-1:1994, 1.2. In particular, these procedures are based on Formulae (2) to (6) of ISO 5725-1:1994, Clause 5.

The model is

$$y = m + B + e$$

where, for the particular material tested,

- m is the general mean (expectation);
- B is the laboratory component of bias under repeatability conditions;
- e is the random error occurring in every measurement under repeatability conditions.

NOTE The laboratory component of bias, B , represents the deviation of a laboratory mean from the general average m .

5.2 ISO 5725-1:1994, Formulae (2) to (6), are expressed in terms of the true standard deviations of the populations considered. In practice, the exact values of these standard deviations are not known, and estimates of precision values must be made from a relatively small sample of all the possible laboratories, and within those laboratories from a small sample of all the possible test results.

5.3 In statistical practice, where the true value of a standard deviation, σ , is not known and is replaced by an estimate based upon a sample, then the symbol σ is replaced by s to denote that it is an estimate. This is done in each of ISO 5725-1:1994, Formulae (2) to (6), giving:

- s_L^2 is the estimate of the between-laboratory variance;
- s_W^2 is the estimate of the within-laboratory variance;
- s_r^2 is the arithmetic mean of s_W^2 and is the estimate of the repeatability variance; this arithmetic mean is taken over all those laboratories taking part in the accuracy experiment which remain after outliers have been excluded;
- s_R^2 is the estimate of the reproducibility variance:

$$s_R^2 = s_L^2 + s_r^2 \quad (1)$$

6 Requirements for a precision experiment

6.1 Layout of the experiment

6.1.1 In the layout used in the basic method, samples from q batches of materials, representing q different levels of the test, are sent to p laboratories which each obtain exactly n replicate test results under repeatability conditions at each of the q levels. This type of experiment is called a balanced uniform-level experiment.

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6.1.2 The performance of these measurements shall be organized and instructions issued as follows.

- a) Any preliminary checking of equipment shall be as specified in the standard method.
- b) Each group of n measurements belonging to one level shall be carried out under repeatability conditions, i.e. within a short interval of time and by the same operator, and without any intermediate recalibration of the apparatus unless this is an integral part of performing a measurement.
- c) It is essential that a group of n tests under repeatability conditions be performed independently as if they were n tests on different materials. As a rule, however, the operator knows that he/she is testing identical material, but the point should be stressed in the instructions that the whole purpose of the experiment is to determine what differences in results can occur in actual testing. If it is feared that, despite this warning, previous results can influence subsequent test results and thus the repeatability variance, it should be considered whether to use n separate samples at each of the q levels, coded in such a way that the operator does not know which are the replicates for a given level. However, such a procedure can cause problems in ensuring that repeatability conditions apply between replicates. This is only possible if the measurements are of such a nature that all the qn measurements can be performed within a short interval of time.
- d) It is not essential that all the q groups of n measurements each be performed strictly within a short interval of time; different groups of measurements may be carried out on different days.
- e) Measurements of all q levels shall be performed by one and the same operator and, in addition, the n measurements at a given level shall be performed using the same equipment throughout.
- f) If in the course of the measurements an operator should become unavailable, another operator may complete the measurements, provided that the change does not occur within a group of n measurements at one level but only occurs between two of the q groups. Any such change shall be reported with the results.

ISO 5725-2:2019(E)

- g) A time limit shall be given within which all measurements shall be completed. This can be necessary to limit the time allowed to elapse between the day the samples are received and the day the measurements are performed.
- h) All samples shall be clearly labelled with the name of the experiment and a sample identification.

6.1.3 In [6.1.2](#) and elsewhere in this document, reference is made to the operator. For some measurements, there can in fact be a team of operators, each of whom performs some specific part of the procedure. In such a case, the team shall be regarded as “the operator” and any change in the team shall be regarded as providing a different “operator”.

6.1.4 In commercial practice, the test results can be rounded rather crudely, but in a precision experiment test results shall be reported to at least one more digit than specified in the standard method. If the method does not specify the number of digits, the rounding shall not be coarser than half the repeatability standard deviation estimate. When precision depends on the level m , different degrees of rounding can be necessary for different levels.

6.2 Recruitment of the laboratories

6.2.1 The general principles regarding recruitment of the laboratories to participate in an interlaboratory experiment are given in ISO 5725-1. Guidance on the number of laboratories is given in [Annex A](#). In enlisting the cooperation of the requisite number of laboratories, their responsibilities shall be clearly stated. An example of a suitable enlistment questionnaire is given in [Figure 1](#).

6.2.2 For the purposes of this document, a “laboratory” is considered to be a combination of the operator, the equipment and the test site. One test site (or laboratory in the conventional sense) can thus produce several “laboratories” if it can provide several operators each with independent sets of equipment and situations in which to perform the work.

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6.3 Preparation of the materials

6.3.1 A discussion of the points that need to be considered when selecting materials for use in a precision experiment is given in ISO 5725-1.

6.3.2 When deciding on the quantities of material to be provided, allowance shall be made for accidental spillage or errors in obtaining some test results which can necessitate using extra material. The amount of material prepared shall be sufficient to cover the experiment and allow an adequate stock in reserve.

6.3.3 It should be considered whether it is desirable for some laboratories to obtain some preliminary test results for familiarization with the measurement method before obtaining the official test result and, if so, whether additional material (not precision experiment samples) should be provided for this purpose.

6.3.4 When a material is to be homogenized, this shall be done in the manner most appropriate for that material. When the material to be tested is not homogeneous, it is important to prepare the samples in the manner specified in the method, preferably starting with one batch of commercial material for each level. In the case of unstable materials, special instructions on storage and treatment shall be specified.

NOTE ISO Guide 35 gives information on evaluating homogeneity and stability for reference materials.

6.3.5 For the samples at each level, n separate containers shall be used for each laboratory if there is any danger of the materials deteriorating once the container has been opened (e.g. by oxidation, by losing volatile components, or with hygroscopic material). In the case of unstable materials, special instructions on storage and treatment shall be specified. Precautions can be necessary to ensure that samples

remain identical up to the time the measurements are made. If the material to be measured consists of a mixture of powders of different relative density or of different grain size, some care is needed because segregation can result from shaking, for example during transport. When reaction with the atmosphere can be expected, the specimens may be sealed into ampoules, either evacuated or filled with an inert gas. For perishable materials such as food or blood samples, it can be necessary to send them in a deep-frozen state to the participating laboratories with detailed instructions for the procedure for thawing.

Questionnaire for interlaboratory study	
Title of measurement method (copy attached)	
1. Our laboratory is willing to participate in the precision experiment for this standard measurement method	
YES <input type="checkbox"/>	NO <input type="checkbox"/> (tick appropriate box)
2. As a participant, we understand that:	
a) all essential apparatus, chemicals, and other requirements specified in the method must be available in our laboratory when the programme begins;	
b) specified "timing" requirements such as starting date, order of testing specimens and finishing date of the programme must be rigidly met;	
c) the method must be strictly adhered to;	
d) samples must be handled in accordance with instructions;	
e) a qualified operator must perform the measurements.	
Having studied the method and having made a fair appraisal of our capabilities and facilities, we feel that we will be adequately prepared for cooperative testing of this method.	
3. <u>Comments</u>	
	(Signed)
	(Company or laboratory)

Figure 1 — Enlistment questionnaire for interlaboratory study

7 Personnel involved in a precision experiment

NOTE The methods of operation within different laboratories are not expected to be identical. Therefore, the contents of this clause are only intended as a guide to be modified as appropriate to cater for a particular situation.

7.1 Panel

7.1.1 The precision experiment should be overseen by a panel which should consist of experts familiar with the measurement method and its application.