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

Part 4:
Basic methods for the determination of the trueness of a standard measurement method

Exactitude (justesse et fidélité) des résultats et méthodes de mesure —

Partie 4: Méthodes de base pour la détermination de la justesse d'une méthode de mesure normalisée

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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, Subcommittee SC 6, *Measurement methods and results*.

This second edition cancels and replaces the first edition (ISO 5725-4:1994), which has been technically revised.

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

— clearly recognizing the requirements of the accepted reference values used in bias evaluation experiments and introducing the uncertainties of the accepted reference values,

— changing examples with a currently used measurement method.

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.

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 expectation of a measurement result and a true value. "Precision" refers to the closeness of agreement between independent measurement results obtained under stipulated conditions.

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 document, because it gives the underlying definitions and general principles.

The “trueness” of a measurement method is of interest when it is possible to conceive of a true value for the property being measured. Although the true value cannot be known exactly, it can be possible to have an accepted reference value for the property being measured; for example, if suitable reference materials or measurement standards are available, or if the accepted reference value can be established by reference to another measurement method or by preparation of a known sample. The trueness of the measurement method can be investigated by comparing the accepted reference value with the level of the results given by the measurement method. Trueness is normally expressed in terms of bias. Bias can arise, for example, in chemical analysis if the measurement method fails to extract all of an element, or if the presence of one element interferes with the determination of another.

Two measures of trueness are of interest and both are considered in this document.

a) Bias of the measurement method: where there is a possibility that the measurement method can give rise to a bias, which persists wherever and whenever the measurement is done, then it is of interest to investigate the “bias of the measurement method”. This requires an experiment involving many laboratories.

b) Laboratory bias: measurements within a single laboratory can reveal the “laboratory bias” (as defined in ISO 5725-1). If it is proposed to undertake an experiment to estimate laboratory bias, then it should be realized that the estimate is valid only at the time of the experiment and at the investigated level(s) for the property. Further regular testing is required to show that the laboratory bias does not vary; the method described in ISO 5725-6 can be used for this.

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

Part 4:
Basic methods for the determination of the trueness of a standard measurement method

1 Scope

1.1 This document

— specifies basic methods for estimating the bias of a measurement method and the laboratory bias when a measurement method is applied;

— provides a practical approach of a basic method for routine use in estimating the bias of measurement methods and laboratory bias;

— provides a brief guidance to all personnel concerned with designing, performing or analysing the results of the measurements for estimating bias.

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

1.3 This document applies when the measurement method has been standardized and all measurements are carried out according to that measurement method.

NOTE In ISO/IEC Guide 99:2007(VIM), “measurement procedure” (2.6) is an analogous term related to the term “measurement method” used in this document.

1.4 This document applies only if an accepted reference value can be established to substitute the true value by using the value, for example:

— of a suitable reference material;

— of a suitable measurement standard;

— referring to a suitable measurement method;

— of a suitable prepared known sample.

1.5 This document applies only to the cases where it is sufficient to estimate bias on one property at a time. It is not applicable if the bias in the measurement of one property is affected by the level of any other property (i.e. it does not consider interferences by any influencing quantity). Comparison of the trueness of two-measurement methods is considered in ISO 5725-6.

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: General statistical terms and terms used in probability*

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

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

ISO 5725-2, *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*

ISO Guide 30, *Reference materials — Selected terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3534-1, ISO 3534-2, ISO 5725-1 and ISO Guide 30 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/

4 Symbols

A	Factor used to calculate the measurement uncertainty of an estimate
B	Laboratory component of bias
C, C', C''	Test statistics
C_{crit}, C'_{crit}, C''_{crit}	Critical values for statistical tests
e	Random error occurring in every measurement under repeatability conditions
G	Grubbs' test statistic
h	Mandel's between-laboratory consistency test statistic
k	Mandel's within-laboratory consistency test statistic
n	Number of measurement results obtained in one laboratory at one level of the property being measured (i.e. per cell)
p	Number of laboratories participating in the interlaboratory experiment
P	Probability
s	Estimate of a standard deviation
u	Standard measurement uncertainty; quantile of the standard normal distribution
y	Measurement result
$\bar{y}$	Average of the measurement results
$\bar{\bar{y}}$	Grand mean of the measurement results
α	Significance level (α is assumed to be 0,05 in this document)

β	Type II error probability
Φ	Cumulative distribution function of the standard normal distribution
γ	Ratio of the reproducibility standard deviation to the repeatability standard deviation (σ_R/σ_r)
δ	Bias of the measurement method under investigation
$\hat{\delta}$	Estimate of the bias of the measurement method under investigation
Δ	Laboratory bias
$\hat{\Delta}$	Estimate of the laboratory bias
μ	Accepted reference value of a property being measured
ν	Number of degrees of freedom
σ	True value of a standard deviation
$\chi_P^2(\nu)$	P-quantile of the χ^2-distribution with ν degrees of freedom

Subscripts

i	Identifier for a participating laboratory; identifier for an individual laboratory (inner laboratory)
k	Identifier for a particular measurement result from a laboratory
L	Between-laboratory (interlaboratory)
m	Identifier for detectable bias
P	Probability
r	Repeatability
R	Reproducibility
y	Identifier for the measurement result
0	Identifier for the accepted reference value

5 Determination of the bias of a standard measurement method by an interlaboratory experiment

5.1 Experimental design considerations

5.1.1 Objective

The objective of the experiment is to estimate the value of the bias of the measurement method and to determine when it is statistically significant. When the bias is found to be statistically insignificant, then the objective is to determine the maximum absolute value of bias that can remain, with a certain probability, undetected by the results of the experiment.

5.1.2 Layout of the experiment

The layout of the experiment is almost the same as that for a precision experiment, as described in ISO 5725-2. The differences are

— the number of participating laboratories and the number of measurement results shall also satisfy the requirements given in 5.3, and

— there is an additional requirement, given in 5.4, to use an accepted reference value of the property being measured.

5.1.3 Cross-references to ISO 5725-1 and ISO 5725-2

Requirements on experimental design given in ISO 5725-1 and ISO 5725-2 apply. When reading ISO 5725-1 and ISO 5725-2 in this context, "trueness" shall be inserted in place of “precision” or “repeatability and reproducibility” as appropriate.

5.2 The statistical model

The basic model of a measurement result, y, can be expressed as

$$y=\mu+\delta+B+e \tag{1}$$

where

μ is the accepted reference value of a property being measured;

δ is the bias of the measurement method under investigation;

B is the laboratory component of bias;

e is the random error occurring in every measurement under repeatability conditions.

NOTE In this document, bias is evaluated at one level at a time; for convenience, the index j, defined in ISO 5725-1, for the level of property has been omitted throughout.

When all of measurement results are obtained according to the requirements in 5.3 and 5.4 from the sufficient number of participant laboratories and sufficient number of measurements under repeatability conditions in each laboratory by using the same measurement method, the bias of the measurement method, at each level of the property, is estimated by

$$\hat{\delta}=\bar{\bar{y}}-\mu \tag{2}$$

where

$\hat{\delta}$ is an estimate of the bias of the measurement method under investigation;

$\bar{\bar{y}}$ is the grand mean of the measurement results from all participant laboratories;

μ is the accepted reference value of the property being measured.

5.3 Required number of laboratories and measurements

The number of laboratories and the number of measurement results required at each laboratory are interdependent. Guidance on selecting these numbers is given below. Although it is assumed that the laboratory biases can be regarded as draws from an approximately normal distribution, in practice the guidance is appropriate for most unimodal distributions.

For the results of an experiment to be able to detect with a high probability a predetermined maximum absolute value of bias, δ_m, the following formula shall be satisfied:

$$A\sigma_R \le \frac{\delta_m}{1,84} \tag{3}$$

where

A is a factor used in calculating the measurement uncertainty of an estimate of bias (see below);

σ_R is the reproducibility standard deviation of the measurement method;

δ_m is the predetermined maximum absolute value of bias that the experimenter wishes to detect from the results of the experiment;

1,84 is a derived factor (see Annex A).

In Formula (3), A is a function of the number of laboratories, the number of measurement results in each laboratory, the reproducibility standard deviation of the measurement method and the measurement uncertainty of the accepted reference value. A is given by

$$A = 1,96\sqrt{A_0^2 + A_y^2} = 1,96\sqrt{\frac{u^2(\mu)}{\sigma_R^2} + \frac{n(\gamma^2 - 1) + 1}{\gamma^2 pn}} \tag{4}$$

where

1,96 is the 0,975-quantile of the standard normal distribution (see Annex A);

A_0 is the ratio of the standard measurement uncertainty of the accepted reference value to the reproducibility standard deviation of the measurement method;

A_y is the ratio of the standard deviation of the grand mean in this experiment to the reproducibility standard deviation of the measurement method;

$u(\mu)$ is the standard measurement uncertainty of the accepted reference value;

n is the number of measurement results in each laboratory;

p is the number of participating laboratories;

γ is the ratio of the reproducibility standard deviation to the repeatability standard deviation.

In Formula (4), A_0, A_y and γ are given respectively by

$$A_0 = u(\mu)/\sigma_R \tag{5}$$

$$A_y = \sqrt{\frac{n(\gamma^2 - 1) + 1}{\gamma^2 pn}} \tag{6}$$

$$\gamma = \sigma_R / \sigma_r \tag{7}$$

where σ_r is the repeatability standard deviation of the measurement method.

If the measurement uncertainty of the accepted reference value is small enough to be neglected, implying $A_0 \leq 0{,}3A_y$ (i.e. $u(\mu) \leq 0{,}3A_y\sigma_R$), Formula (4) may be simplified to

$$A = 1{,}96A_y \tag{8}$$

The values of A calculated by Formula (8) are given in Table 1.

Ideally, the choice of the combination of the number of laboratories and the number of replicate measurement results per laboratory should satisfy the requirement described by Formula (3), with the δ_m value predetermined by the experimenter. However, for practical reasons, the choice of the number of laboratories is usually a compromise between the availability of resources and the desire to reduce the value of δ_m to a satisfactory extent. If the reproducibility of the measurement method is poor, then it is not practical to achieve a high degree of certainty in the estimate of the bias. When σ_R is larger than σ_r (i.e. γ is larger than 1) as is often the case, little is to be gained by obtaining more than $n = 2$ measurement results per laboratory per level.

Table 1 — Values of A, the factor used in calculating the measurement uncertainty of an estimate of bias in the case when the measurement uncertainty of the accepted reference value is small enough to be neglected

No. of laboratories	Value of A calculated by Formula (8)								
	$\gamma = 1$			$\gamma = 2$			$\gamma = 5$		
p	$n = 2$	$n = 3$	$n = 4$	$n = 2$	$n = 3$	$n = 4$	$n = 2$	$n = 3$	$n = 4$
5	0,62	0,51	0,44	0,82	0,80	0,79	0,87	0,86	0,86
10	0,44	0,36	0,31	0,58	0,57	0,56	0,61	0,61	0,61
15	0,36	0,29	0,25	0,47	0,46	0,46	0,50	0,50	0,50
20	0,31	0,25	0,22	0,41	0,40	0,40	0,43	0,43	0,43
25	0,28	0,23	0,20	0,37	0,36	0,35	0,39	0,39	0,39
30	0,25	0,21	0,18	0,33	0,33	0,32	0,35	0,35	0,35
35	0,23	0,19	0,17	0,31	0,30	0,30	0,33	0,33	0,33
40	0,22	0,18	0,15	0,29	0,28	0,28	0,31	0,31	0,31

5.4 Requirements of the accepted reference value

5.4.1 Approaches to assigning the accepted reference value

The accepted reference value of the property of interest, μ, shall be reliable and metrologically traceable to an accepted reference by which the true value can be substituted. It refers to the value carried by the material used in this experiment, which was either assigned in another independent study, such as the characterization of the reference material, the calibration of the measurement standard by using suitable calibration procedures and competent laboratories, the assignment by other measurement method (preferably a reference measurement method), or calculated from the property values of the materials used for preparation of the known sample.

NOTE Guidance for the characterization and use of reference materials is given in ISO Guide 35 and ISO Guide 33, respectively. Refer to the definition for other measurement standards in ISO/IEC Guide 99 (VIM).

5.4.2 Materials used in the experiment

5.4.2.1 The material used in the experiment, whether purchased or prepared, can be a reference material, a measurement standard or a prepared known sample; it shall have the same property as that the standard measurement method is intended to be applied to, e.g. concentration.

5.4.2.2 The value of the property of interest carried by the material, which has been assigned by any approach listed in 5.4.1, shall be appropriate to the range of values at which the standard measurement

