

STN	Presnosť (správnosť a zhodnosť) metód a výsledkov merania Časť 3: Medzil'ahlá zhodnosť a alternatívne návrhy pre spolupracujúce štúdie	STN ISO 5725-3 01 0251
------------	---	--

Accuracy (trueness and precision) of measurement methods and results

Part 3: Intermediate precision and alternative designs for collaborative studies

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

Partie 3: Fidélité intermédiaire et plans alternatifs pour les études collaboratives

Táto slovenská technická norma obsahuje anglickú verziu medzinárodnej normy ISO 5725-3: 2023 a má postavenie oficiálnej verzie.

This Slovak standard includes the English version of the International standard ISO 5725-3: 2023 and has the status of the official version.

139750

Úrad pre normalizáciu, metrológiu a skúšobníctvo Slovenskej republiky, 2025

Slovenská technická norma a technická normalizačná informácia je chránená zákonom č. 60/2018 Z. z. o technickej normalizácii v znení neskorších predpisov.

Anotácia

Tento dokument poskytuje

- diskusiu o alternatívnych návrhoch experimentov na určenie správnosti a zhodnosti meraní vrátane reprodukovateľnosti, opakovateľnosti a vybraných mier medziľahlej zhodnosti normalizovanej metódy merania, vrátane preskúmania okolností, za ktorých je ich použitie nevyhnutné alebo prospešné a návod ako na interpretáciu a aplikáciu výsledných odhadov; a
- spracované príklady vrátane konkrétnych návrhov a výpočtov.

Národný predhovor

Normatívne referenčné dokumenty

Na nasledujúce dokumenty sa odkazuje v texte takým spôsobom, že časť ich obsahu alebo celý obsah predstavuje požiadavky tohto dokumentu. Pri datovaných odkazoch sa používa len citované vydanie. Pri nedatovaných odkazoch sa používa najnovšie vydanie citovaného dokumentu (vrátane akýchkoľvek zmien).

POZNÁMKA 1. – Ak bola medzinárodná publikácia zmenená spoločnými modifikáciami, čo je indikované označením (mod), použije sa príslušná EN/HD.

POZNÁMKA 2. – Aktuálne informácie o platných a zrušených STN a TNI možno získať na webovom sídle www.unms.sk.

ISO 3534-1 prijatá ako STN ISO 3534-1 Štatistika. Slovník a značky. Časť 1: Všeobecné štatistické termíny a termíny používané v teórii pravdepodobnosti (01 0216)

ISO 3534-2 prijatá ako STN ISO 3534-2 Štatistika. Slovník a značky. Časť 2: Aplikovaná štatistika (01 0216)

ISO 5725-1 prijatá ako STN ISO 5725-1 Presnosť (správnosť a zhodnosť) metód a výsledkov merania. Časť 1: Všeobecné zásady a definície (01 0251)

ISO Guide 33 dosiaľ neprijatý

ISO Guide 35 dosiaľ neprijatý

Vypracovanie

Spracovateľ: Úrad pre normalizáciu, metrológiu a skúšobníctvo Slovenskej republiky, Bratislava

Technická komisia: TK 71 Aplikácie štatistických metód

Contents

Page

Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	2
3 Terms and definitions	2
4 Symbols	3
5 General requirements	4
6 Intermediate measures of the precision of a standard measurement method	5
6.1 Factors and factor levels	5
6.1.1 Definitions and examples	5
6.1.2 Selection of factors of interest	6
6.1.3 Random and fixed effects	6
6.1.4 Statistical model	7
6.2 Within-laboratory study and analysis of intermediate precision measures	9
6.2.1 Simplest approach	9
6.2.2 Alternative method	10
6.2.3 Effect of the measurement conditions on the final quoted result	10
7 Nested design	11
7.1 Balanced fully-nested design	11
7.2 Staggered-nested design	12
7.3 Balanced partially-nested design	13
7.4 Orthogonal array design	14
8 Design for heterogeneous material	16
8.1 Applications of the design for a heterogeneous material	16
8.2 Layout of the design for a heterogeneous material	17
8.3 Statistical analysis	17
9 Split-level design	17
9.1 Applications of the split-level design	17
9.2 Layout of the split-level design	19
9.3 Statistical analysis	19
10 Design across levels	19
10.1 Applications of the design across levels	19
10.2 Layout of the design across levels	20
10.3 Statistical analysis	20
11 Reliability of interlaboratory parameters	20
11.1 Reliability of precision estimates	20
11.2 Reliability of estimates of the overall mean	21
11.2.1 General	21
11.2.2 Balanced fully-nested design (2 factors)	21
11.2.3 Staggered nested design (2 factors)	21
11.2.4 Balanced partially-nested design	21
11.2.5 Orthogonal array design	21
11.2.6 Split-level design	22
Annex A (informative) Fully- and partially-nested designs	23
Annex B (informative) Analysis of variance for balanced fully-nested design	25
Annex C (informative) Analysis of variance for staggered design	30
Annex D (informative) Analysis of variance for the balanced partially-nested design (three factors)	38

ISO 5725-3:2023(E)

Annex E (informative) Statistical model for an experiment with heterogeneous material	41
Annex F (informative) Analysis of variance for split-level design	42
Annex G (informative) Example for split-level design	44
Annex H (informative) Design across levels	47
Annex I (informative) Restricted maximum likelihood (REML)	48
Annex J (informative) Examples of the statistical analysis of intermediate precision experiment	49
Annex K (informative) Example for an analysis across levels	55
Bibliography	57

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 document 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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 5725-3:1994), which has been technically revised. It also incorporates the Technical Corrigendum ISO 5725-3:1994/Cor.1:2001.

The main changes are as follows:

- Several additional experimental designs have been added to this version compared to the previous version, some of them from ISO 5725-5. These are orthogonal array designs, split level designs, designs for heterogeneous sample material as well as designs across levels.
- Furthermore, the standard was supplemented by considerations on the selection of factors and modelling of the factorial effects, as well as by a section in which the reliability of the various interlaboratory test parameters (mean and precision parameters) are considered.

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 5725-3:2023(E)

Introduction

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

0.2 General consideration of these quantities is given in ISO 5725-1 and is not repeated here. It is stressed that ISO 5725-1 provides underlying definitions and general principles should be read in conjunction with all other parts of ISO 5725.

0.3 Many different factors (apart from test material heterogeneity) may contribute to the variability of results from a measurement method, including:

- a) the laboratory;
- b) the operator;
- c) the equipment used;
- d) the calibration of the equipment;
- e) the batch of a reagent;
- f) the time elapsed between measurements;
- g) environment (temperature, humidity, air pollution, etc.);
- h) other factors.

0.4 Two conditions of precision, termed repeatability and reproducibility conditions, have been found necessary and, for many practical cases, useful for describing the variability of a measurement method. Under repeatability conditions, none of the factors a) to h) in 0.3 are considered to vary, while under reproducibility conditions, all of the factors are considered to vary and contribute to the variability of the test results. Thus, repeatability and reproducibility conditions are the two extremes of precision, the first describing the minimum and the second the maximum variability in results. Intermediate conditions between these two extreme conditions of precision are also conceivable, when one or more of the factors listed in b) to g) are allowed to vary.

To illustrate the need for including a consideration of intermediate conditions in method validation, consider the operation of a present-day laboratory connected with a production plant involving, for example, a three-shift working system where measurements are made by different operators on different equipment. Operators and equipment are then some of the factors that contribute to the variability in the test results.

The standard deviation of test results obtained under repeatability conditions is generally less than that obtained under intermediate precision conditions. Generally, in chemical analysis, the standard deviation under intermediate precision conditions may be two or three times larger than that under repeatability conditions. It should not, of course, exceed the reproducibility standard deviation.

As an example, in the determination of copper in copper ore, a collaborative study among 35 laboratories revealed that the standard deviation under intermediate precision conditions (different times) was 1,5 times larger than that under repeatability conditions, both for the electrolytic gravimetry and $\text{Na}_2\text{S}_2\text{O}_3$ titration methods.

0.5 This document focuses on intermediate precision and alternative designs for collaborative studies of a measurement method. Apart from the determination of intermediate precision measures, the aims of these alternative designs include reducing the number of required measurements, increasing the reliability of the estimates for precision and overall mean and taking into account test material heterogeneity.

Indeed, a t -factor fully-nested experiment with two levels per factor (inside each laboratory, there are $t-1$ factors) and two replicates per setting requires $2 \cdot 2^{t-1}$ test results from each laboratory, which can be an excessive requirement on the laboratories. For this reason, in the previous version of ISO 5725-3, the staggered nested design is also discussed. While the estimation of the precision parameters is more complex and subject to greater uncertainty in a staggered nested design, the workload is reduced. This document offers alternative strategies to reduce the workload without compromising the reliability of the precision estimates.

As far as the special designs for sample heterogeneity are concerned, they were discussed in the previous version of ISO 5725-5. However, it is convenient to have one part of this standard dedicated to the question of the design of experiments.

0.6 The repeatability precision as determined in accordance with ISO 5725-2 is computed as a mean across participating laboratories. Whether it can be used for quality control purposes depends on whether the repeatability standard deviation can be considered to remain constant across laboratories. For this reason, it is important to obtain information on how the repeatability standard deviation varies within and between the laboratories under different conditions.

0.7 In many collaborative studies, the between-laboratory variability is large in comparison to the repeatability, and it would be useful to a) decompose it into several different precision components, b) reduce, if possible, some sources of variability which are due to the intermediate precision conditions. This can be done by identifying factors (e.g. time, calibration, operator or equipment) which contribute to the variability under intermediate precision conditions of measurement, by quantifying the corresponding variability components and, wherever achievable, decreasing their contribution. In this manner, the intermediate precision component of the overall variance is enlarged while the between-laboratory component of the overall variance is reduced. Only random effects are considered: it is only reasonable to model a factor as a fixed effect after a method or calibration optimization study has been conducted. In this standard, different relationships between factors are taken into account, e.g. whether a particular factor is subsumed under another factor or not.

0.8 Estimates for precision and overall mean are subject to random variability. Accordingly, it is important to determine the uncertainty associated with each estimate, and to understand the relationships between this uncertainty, the number of participants and the design. Once these relationships are understood, it becomes possible to make much more informed decisions concerning the number of participants and the experimental design.

0.9 Provided different factorial effects do contribute to the variability, determining the respective precision components may make it possible to reduce the required number of participating laboratories, since the between-laboratory variability can be expected to be less dominant. However, it is highly recommended to have a reasonable number of participating laboratories in order to ensure a realistic assessment of the overall method variability obtained under routine conditions of operation.

0.10 In the uniform-level design according to part 2 of this standard, there is a risk that an operator will allow the result of a measurement on one sample to influence the result of a subsequent measurement on another sample of the same material, causing the estimates of the repeatability and reproducibility standard deviations to be biased. When this risk is considered to be serious, the split-level design described in this document may be preferred as it reduces this risk. Care should be taken that the two materials used at a particular level of the experiment are sufficiently similar to ensure that the same precision measures can be expected (in other words: the question arises whether the precision component associated with a particular factor remains unchanged across a range of similar matrices).

0.11 The experimental design presented in ISO 5725-2 requires the preparation of a number of identical samples of the material for use in the experiment. With heterogeneous materials this may not be possible, so that the use of the basic method then gives estimates of the reproducibility standard deviation that are inflated by the variation between the samples. The design for a heterogeneous material given in this document yields information about the variability between samples which is not obtainable from the basic method; it may be used to calculate an estimate of reproducibility from which the between-sample variation has been removed.

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

Part 3: Intermediate precision and alternative designs for collaborative studies

1 Scope

This document provides

- a) a discussion of alternative experimental designs for the determination of trueness and precision measures including reproducibility, repeatability and selected measures of intermediate precision of a standard measurement method, including a review of the circumstances in which their use is necessary or beneficial, and guidance as to the interpretation and application of the resulting estimates, and
- b) worked examples including specific designs and computations.

Each of the alternative designs discussed in this document is intended to address one (or several) of the following issues:

- a) a discussion of the implications of the definitions of intermediate precision measures;
- b) a guidance on the interpretation and application of the estimates of intermediate precision measures in practical situations;
- c) determining reproducibility, repeatability and selected measures of intermediate precision;
- d) improved¹⁾ determination of reproducibility and other measures of precision;
- e) improving the estimate of the sample mean;
- f) determining the range of in-house repeatability standard deviations;
- g) determining other precision components such as operator variability;
- h) determining the level of reliability of precision estimates;
- i) reducing the minimum number of participating laboratories by optimizing the reliability of precision estimates;
- j) avoiding distorted estimations of repeatability (split-level designs);
- k) avoiding distorted estimations of reproducibility (taking the heterogeneity of the material into consideration).

Often, the performance of the method whose precision is being evaluated in a collaborative study will have previously been assessed in a single-laboratory validation study conducted by the laboratory which developed it. Relevant factors for the determination of intermediary precision will have been identified in this prior single-laboratory study.

1) Allowing a reduction in the number of laboratories.

ISO 5725-3:2023(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: 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 Guide 33, *Reference materials — Good practice in using reference materials*

ISO Guide 35, *Reference materials — Guidance for characterization and assessment of homogeneity and stability*

koniec náhľadu – text ďalej pokračuje v platenej verzii STN