

INTERNATIONAL STANDARD

ISO
5725-3

First edition
1994-12-15

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

Part 3:

Intermediate measures of the precision of a
standard measurement method

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

*Partie 3: Mesures intermédiaires de la fidélité d'une méthode de mesure
normalisée*



Reference number
ISO 5725-3:1994(E)

Contents

Page

1	Scope	1
2	Normative references	2
3	Definitions	2
4	General requirement	2
5	Important factors	2
6	Statistical model	3
6.1	Basic model	3
6.2	General mean, m	3
6.3	Term B	4
6.4	Terms B_0, $B_{(1)}$, $B_{(2)}$, etc.	4
6.5	Error term, e	5
7	Choice of measurement conditions	5
8	Within-laboratory study and analysis of intermediate precision measures	6
8.1	Simplest approach	6
8.2	An alternative method	6
8.3	Effect of the measurement conditions on the final quoted result	7
9	Interlaboratory study and analysis of intermediate precision measures	7
9.1	Underlying assumptions	7
9.2	Simplest approach	7
9.3	Nested experiments	7
9.4	Fully-nested experiment	8
9.5	Staggered-nested experiment	9
9.6	Allocation of factors in a nested experimental design	9

© ISO 1994

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

9.7	Comparison of the nested design with the procedure given in ISO 5725-2	9
9.8	Comparison of fully-nested and staggered-nested experimental designs	9

Annexes

A	Symbols and abbreviations used in ISO 5725	10
B	Analysis of variance for fully-nested experiments	12
B.1	Three-factor fully-nested experiment	12
B.2	Four-factor fully-nested experiment	13
C	Analysis of variance for staggered-nested experiments	15
C.1	Three-factor staggered-nested experiment	15
C.2	Four-factor staggered-nested experiment	16
C.3	Five-factor staggered-nested experiment	17
C.4	Six-factor staggered-nested experiment	18
D	Examples of the statistical analysis of intermediate precision experiments	19
D.1	Example 1 — Obtaining the [time + operator]-different intermediate precision standard deviation, $s_{1(\tau\theta)}$, within a specific laboratory at a particular level of the test	19
D.2	Example 2 — Obtaining the time-different intermediate precision standard deviation by interlaboratory experiment	20
E	Bibliography	25

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 5725-3 was prepared by Technical Committee ISO/TC 69, *Applications of statistical methods*, Subcommittee SC 6, *Measurement methods and results*.

ISO 5725 consists of the following parts, under the general title *Accuracy (trueness and precision) of measurement methods and results*:

- *Part 1: General principles and definitions*
- *Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*
- *Part 3: Intermediate measures of the precision of a standard measurement method*
- *Part 4: Basic methods for the determination of the trueness of a standard measurement method*
- *Part 5: Alternative methods for the determination of the precision of a standard measurement method*
- *Part 6: Use in practice of accuracy values*

Parts 1 to 6 of ISO 5725 together cancel and replace ISO 5725:1986, which has been extended to cover trueness (in addition to precision) and intermediate precision conditions (in addition to repeatability conditions and reproducibility conditions).

Annexes A, B and C form an integral part of this part of ISO 5725. Annexes D and E are for information only.

Introduction

0.1 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 average value of a large number of test results and the true or accepted reference value. "Precision" refers to the closeness of agreement between test results.

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

0.3 Many different factors (apart from variations between supposedly identical specimens) may contribute to the variability of results from a measurement method, including:

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

The variability between measurements performed by different operators and/or with different equipment will usually be greater than the variability between measurements carried out within a short interval of time by a single operator using the same equipment.

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, factors a) to f) in 0.3 are considered constants and do not contribute to the variability, while under reproducibility conditions they vary and do 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 factors

a) to f) are allowed to vary, and are used in certain specified circumstances.

Precision is normally expressed in terms of standard deviations.

0.5 This part of ISO 5725 focuses on intermediate precision measures of a measurement method. Such measures are called intermediate as their magnitude lies between the two extreme measures of the precision of a measurement method: repeatability and reproducibility standard deviations.

To illustrate the need for such intermediate precision measures, 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. These factors need to be taken into account when assessing the precision of the measurement method.

0.6 The intermediate precision measures defined in this part of ISO 5725 are primarily useful when their estimation is part of a procedure that aims at developing, standardizing, or controlling a measurement method within a laboratory. These measures can also be estimated in a specially designed interlaboratory study, but their interpretation and application then requires caution for reasons explained in 1.3 and 9.1.

0.7 The four factors most likely to influence the precision of a measurement method are the following.

- a) **Time:** whether the time interval between successive measurements is short or long.
- b) **Calibration:** whether the same equipment is or is not recalibrated between successive groups of measurements.
- c) **Operator:** whether the same or different operators carry out the successive measurements.
- d) **Equipment:** whether the same or different equipment (or the same or different batches of reagents) is used in the measurements.

0.8 It is, therefore, advantageous to introduce the following M -factor-different intermediate precision conditions ($M = 1, 2, 3$ or 4) to take account of changes in measurement conditions (time, calibration, operator and equipment) within a laboratory.

- a) $M = 1$: only one of the four factors is different;
- b) $M = 2$: two of the four factors are different;
- c) $M = 3$: three of the four factors are different;
- d) $M = 4$: all four factors are different.

Different intermediate precision conditions lead to different intermediate precision standard deviations denoted by $s_{i()}$, where the specific conditions are listed within the parentheses. For example, $s_{i(TO)}$ is the inter-

mediate precision standard deviation with different times (T) and operators (O).

0.9 For measurements under intermediate precision conditions, one or more of the factors listed in 0.7 is or are different. Under repeatability conditions, those factors are assumed to be constant.

The standard deviation of test results obtained under repeatability conditions is generally less than that obtained under the conditions for intermediate precision conditions. Generally in chemical analysis, the standard deviation under intermediate precision conditions may be two or three times as large as 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 experiment among 35 laboratories revealed that the standard deviation under one-factor-different intermediate precision conditions (operator and equipment the same but time different) 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.

This document is a preview generated by EVS

This document is a preview generated by EVS

This page intentionally left blank

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

Part 3:

Intermediate measures of the precision of a standard measurement method

1 Scope

1.1 This part of ISO 5725 specifies four intermediate precision measures due to changes in observation conditions (time, calibration, operator and equipment) within a laboratory. These intermediate measures can be established by an experiment within a specific laboratory or by an interlaboratory experiment.

Furthermore, this part of ISO 5725

- a) discusses the implications of the definitions of intermediate precision measures;
- b) presents guidance on the interpretation and application of the estimates of intermediate precision measures in practical situations;
- c) does not provide any measure of the errors in estimating intermediate precision measures;
- d) does not concern itself with determining the trueness of the measurement method itself, but does discuss the connections between trueness and measurement conditions.

1.2 This part of ISO 5725 is concerned exclusively with measurement methods which yield measurements on a continuous scale and give a single value as the test result, although the single value may be

the outcome of a calculation from a set of observations.

1.3 The essence of the determination of these intermediate precision measures is that they measure the ability of the measurement method to repeat test results under the defined conditions.

1.4 The statistical methods developed in this part of ISO 5725 rely on the premise that one can pool information from "similar" measurement conditions to obtain more accurate information on the intermediate precision measures. This premise is a powerful one as long as what is claimed as "similar" is indeed "similar". But it is very difficult for this premise to hold when intermediate precision measures are estimated from an interlaboratory study. For example, controlling the effect of "time" or of "operator" across laboratories in such a way that they are "similar", so that pooling information from different laboratories makes sense, is very difficult. Thus, using results from interlaboratory studies on intermediate precision measures requires caution. Within-laboratory studies also rely on this premise, but in such studies it is more likely to be realistic, because the control and knowledge of the actual effect of a factor is then more within reach of the analyst.

1.5 There exist other techniques besides the ones described in this part of ISO 5725 to estimate and to verify intermediate precision measures within a lab-

oratory, for example, control charts (see ISO 5725-6). This part of ISO 5725 does not claim to describe the only approach to the estimation of intermediate precision measures within a specific laboratory.

NOTE 1 This part of ISO 5725 refers to designs of experiments such as nested designs. Some basic information is given in annexes B and C. Other references in this area are given in annex E.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 5725. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 5725 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3534-1:1993, *Statistics — Vocabulary and symbols — Part 1: Probability and general statistical terms*.

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*.

ISO 5725-2:1994, *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 33:1989, *Uses of certified reference materials*.

ISO Guide 35:1989, *Certification of reference materials — General and statistical principles*.

3 Definitions

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

The symbols used in ISO 5725 are given in annex A.

4 General requirement

In order that the measurements are made in the same way, the measurement method shall have been standardized. All measurements forming part of an experiment within a specific laboratory or of an inter-laboratory experiment shall be carried out according to that standard.

5 Important factors

5.1 Four factors (time, calibration, operator and equipment) in the measurement conditions within a laboratory are considered to make the main contributions to the variability of measurements (see table 1).

5.2 "Measurements made at the same time" include those conducted in as short a time as feasible in order to minimize changes in conditions, such as environmental conditions, which cannot always be guaranteed constant. "Measurements made at different times", that is those carried out at long intervals of time, may include effects due to changes in environmental conditions.

Table 1 — Four important factors and their states

Factor	Measurement conditions within a laboratory	
	State 1 (same)	State 2 (different)
Time	Measurements made at the same time	Measurements made at different times
Calibration	No calibration between measurements	Calibration carried out between measurements
Operator	Same operator	Different operators
Equipment	Same equipment without recalibration	Different equipment