TECHNICAL SPECIFICATION

First edition 2005-02-15

Corrected version 2005-07-15

Measurement uncertainty for metrological applications — Repeated measurements and nested experiments

Incertitude de mesure pour les applications en métrologie — Mesures répétées et expériences emboîtées

iTeh STANDARD PREVIEW (standards.iteh.ai)

<u>ISO/TS 21749:2005</u> https://standards.iteh.ai/catalog/standards/sist/218c8386-79cf-4b05-84a5-8ff0e60de301/iso-ts-21749-2005



Reference number ISO/TS 21749:2005(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW (standards.iteh.ai)

ISO/TS 21749:2005

https://standards.iteh.ai/catalog/standards/sist/218c8386-79cf-4b05-84a5-8ff0e60de301/iso-ts-21749-2005

© ISO 2005

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 either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office Case postale 56 • CH-1211 Geneva 20 Tel. + 41 22 749 01 11 Fax + 41 22 749 09 47 E-mail copyright@iso.org Web www.iso.org Published in Switzerland

Contents

Forewordiv		
Introductionv		
1	Scope	1
2	Normative references	1
3	Terms and definitions	2
4 4.1 4.2 4.3 4.4	Statistical methods of uncertainty evaluation Approach of the <i>Guide to the expression of uncertainty of measurement</i> Check standards Steps in uncertainty evaluation Examples in this Technical Specification	3 3 4 5 6
5 5.1 5.2 5.3 5.4 5.5	Type A evaluation of uncertainty	6 7 4 6 7
6	Type B evaluation of uncertainty tandards iteh.ai)	:6
7 7.1 7.2 7.3	Propagation of uncertainty	7 7 8
8 8.1 8.2 8.3 8.4 8.5 8.6	Example — Type A evaluation of uncertainty from a gauge study	0001135
Annex	Annex A (normative) Symbols	
Bibliog	3ibliography	

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

- an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote; NDARD PREVIEW
- an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

ISO/TS 21749:2005

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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.

ISO/TS 21749 was prepared by Technical Committee ISO/TC 69, *Applications of statistical methods*, Subcommittee SC 6, *Measurement methods and results*.

This corrected version of ISO/TS 21749:2005 incorporates the correction of the title.

Introduction

Test, calibration and other laboratories are frequently required to report the results of measurements and the associated uncertainties. Evaluation of uncertainty is an on-going process that can consume time and resources. In particular, there are many tests and other operations carried out by laboratories where two or three sources of uncertainty are involved. Following the approach in the *Guide to the expression of uncertainty of measurement (GUM)* to combining components of uncertainty, this document focuses on using the analysis of variance (ANOVA) for estimating individual components, particularly those based on Type A (statistical) evaluations.

An experiment is designed by the laboratory to enable an adequate number of measurements to be made, the analysis of which will permit the separation of the uncertainty components. The experiment, in terms of design and execution, and the subsequent analysis and uncertainty evaluation, require familiarity with data analysis techniques, particularly statistical analysis. Therefore, it is important for laboratory personnel to be aware of the resources required and to plan the necessary data collection and analysis.

In this Technical Specification, the uncertainty components based on Type A evaluations can be estimated from statistical analysis of repeated measurements, from instruments, test items or check standards.

A purpose of this Technical Specification is to provide guidance on the evaluation of the uncertainties associated with the measurement of test items, for instance as part of ongoing manufacturing inspection. Such uncertainties contain contributions from the measurement process itself and from the variability of the manufacturing process. Both types of contribution include those from operators, environmental conditions and other effects. In order to assist in separating the effects of the measurement process and manufacturing variability, measurements of check standards are used to provide data on the measurement process itself. Such measurements are nominally identical to those made on the test items. In particular, measurements on check standards are used to help identify time-dependent effects, so that such effects can be evaluated and contrasted with a database of check standard measurements. These standards are also useful in helping to control the bias and long-term drift of the process once a baseline for these quantities has been established from historical data.

Clause 4 briefly describes the statistical methods of uncertainty evaluation including the approach recommended in the *GUM*, the use of check standards, the steps in uncertainty evaluation and the examples in this Technical Specification. Clause 5, the main part of this Technical Specification, discusses the Type A evaluations. Nested designs in ANOVA are used in dealing with time-dependent sources of uncertainty. Other sources such as those from the measurement configuration, material inhomogeneity, and the bias due to measurement configurations and related uncertainty analyses are discussed. Type B (non-statistical) evaluations of uncertainty are discussed for completeness in Clause 6. The law of propagation of uncertainty described in the *GUM* has been widely used. Clause 7 provides formulae obtained by applying this law to certain functions of one and two variables. In Clause 8, as an example, a Type A evaluation of uncertainty for a gauge study is discussed, where uncertainty components from various sources are obtained. Annex A lists the statistical symbols used in this Technical Specification.

iTeh STANDARD PREVIEW (standards.iteh.ai)

<u>ISO/TS 21749:2005</u> https://standards.iteh.ai/catalog/standards/sist/218c8386-79cf-4b05-84a5-8ff0e60de301/iso-ts-21749-2005

Measurement uncertainty for metrological applications — Repeated measurements and nested experiments

1 Scope

This Technical Specification follows the approach taken in the *Guide to the expression of the uncertainty of measurement (GUM)* and establishes the basic structure for stating and combining components of uncertainty. To this basic structure, it adds a statistical framework using the analysis of variance (ANOVA) for estimating individual components, particularly those classified as Type A evaluations of uncertainty, i.e. based on the use of statistical methods. A short description of Type B evaluations of uncertainty (non-statistical) is included for completeness.

This Technical Specification covers experimental situations where the components of uncertainty can be estimated from statistical analysis of repeated measurements, instruments, test items or check standards.

It provides methods for obtaining uncertainties from single-, two- and three-level nested designs only. More complicated experimental situations where, for example, there is interaction between operator effects and instrument effects or a cross effect, are not covered.

This Technical Specification is not applicable to measurements that cannot be replicated, such as destructive measurements or measurements on dynamically varying systems (such as fluid flow, electronic currents or telecommunications systems). It is not particularly directed to the certification of reference materials (particularly chemical substances) and to calibrations where artifacts are compared using a scheme known as a "weighing design". For certification of reference materials, see ISO Guide 35^[14].

When results from interlaboratory studies can be used, techniques are presented in the companion guide ISO/TS 21748^[15]. The main difference between ISO/TS 21748 and this Technical Specification is that the ISO/TS 21748 is concerned with reproducibility data (with the inevitable repeatability effects), whereas this Technical Specification concentrates on repeatability data and the use of the analysis of variance for its treatment.

This Technical Specification is applicable to a wide variety of measurements, for example, lengths, angles, voltages, resistances, masses and densities.

2 Normative references

The following referenced documents are indispensable for the application 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 3534-1:1993, Statistics — Vocabulary and symbols — Part 1: Probability and general statistical terms

ISO 3534-3:1999, 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

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 5725-3, Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method

ISO 5725-4, Accuracy (trueness and precision) of measurement methods and results — Part 4: Basic methods for the determination of the trueness of a standard measurement method

ISO 5725-5, Accuracy (trueness and precision) of measurement methods and results — Part 5: Alternative methods for the determination of the precision of a standard measurement method

ISO 5725-6, Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values

Guide to the expression of uncertainty in measurement (GUM), BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML, 1993, corrected and reprinted in 1995

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3534-1, ISO 3534-3, ISO 5725 (all parts) and the following apply.

3.1

3.2

measurand

well-defined physical quantity that is to be measured and can be characterized by an essentially unique value

iTeh STANDARD PREVIEW

uncertainty of measurement

parameter or an estimate of the parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the quantity being measured

ISO/TS 21749:2005

3.3 https://standards.iteh.ai/catalog/standards/sist/218c8386-79cf-4b05-84a5-

Type A evaluation 8ff0e60de301/iso-ts-21749-2005

method of evaluation of uncertainty by using statistical methods

3.4

Type B evaluation

method of evaluation of uncertainty by means other than statistical methods

3.5

standard uncertainty

uncertainty expressed as a standard deviation associated with a single component of uncertainty

3.6

combined standard uncertainty

standard deviation associated with the result of a particular measurement or series of measurements that takes into account one or more components of uncertainty

3.7

expanded uncertainty

combined standard uncertainty multiplied by a coverage factor which usually is an appropriate critical value from the *t*-distribution which depends upon the degrees of freedom in the combined standard uncertainty and the desired level of coverage

3.8

effective degrees of freedom

degrees of freedom associated with a standard deviation composed of two or more components of variance

NOTE The effective degrees of freedom can be computed using the Welch-Satterthwaite approximation (see GUM, G.4).

3.9

nested design

experimental design in which each level (i.e. each potential setting, value or assignment of a factor) of a given factor appears in only a single level of any other factor

NOTE 1 Adapted from ISO 3534-3:1999, definition 2.6.

NOTE 2 See ISO 3434-3:1999, 1.6, for the definition of level.

3.10

fixed effects

(factors) effects resulting from the preselection of levels of each factor over the range of values of the factors

3.11

random effects

 $\langle \text{factors} \rangle$ effects resulting from the sampling at each level of each factor from the population of levels of each factor

3.12

balanced nested design

nested design experiment in which the number of levels of the nested factors is constant

[ISO 3534-3:1999, definition 2.6.1]

3.13

mean square for random errors STANDARD PREVIEW sum of squared error divided by the corresponding degrees of freedom

(standards.iteh.ai

NOTE See ISO 3534-1:1993, 2.85 for the definition of the degrees of freedom.

ISO/TS 21749:2005

https://standards.iteh.ai/catalog/standards/sist/218c8386-79cf-4b05-84a5-

4 Statistical methods of uncertainty evaluation 005

4.1 Approach of the Guide to the expression of uncertainty of measurement

The *Guide to the expression of uncertainty of measurement (GUM)* recommends that the result of measurement be corrected for all recognized significant systematic effects, that the result accordingly be the best (or at least unbiased) estimate of the measurand and that a complete model of the measurement system exists. The model provides a functional relationship between a set of input quantities (upon which the measurand depends) and the measurand (output quantities). The objective of uncertainty evaluation is to determine an interval that can be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand. Since a bias cannot be quantified exactly, when a result of measurement is corrected for bias, the correction has an associated uncertainty.

The general approach, beginning from the modelling process, is the following.

NOTE The approach here relates to input quantities that are mutually independent. It is capable of a further generalization to mutually dependent input quantities (see the *GUM*, 5.2).

- a) Develop a mathematical model (functional relationship) of the measurement process or measurement system that relates the model input quantities (including influence quantities) to the model output quantity (measurand). In many cases, this model is the formula (or formulae) used to calculate the measurement result, augmented if necessary by random, environmental and other effects such as bias correction that may affect the measurement result.
- b) Assign best estimates and the associated standard uncertainties (uncertainties expressed as standard deviations) to the model input quantities.

- Evaluate the contribution to the standard uncertainty associated with the measurement result that is C) attributable to each input quantity. These contributions shall take into account uncertainties associated with both random and systematic effects relating to the input guantities, and may themselves involve more detailed uncertainty evaluations.
- d) Aggregate these standard uncertainties to obtain the (combined) standard uncertainty associated with the measurement result. This evaluation of uncertainty is carried out, according to GUM, using the law of propagation of uncertainty, or by more general analytical or numerical methods when the conditions for the law of propagation of uncertainty do not apply or it is not known whether they apply.
- e) Where appropriate, multiply the standard uncertainty associated with the measurement result by a coverage factor to obtain an expanded uncertainty and hence a coverage interval for the measurand at a prescribed level of confidence. The GUM provides an approach that can be used to calculate the coverage factor. If the degrees of freedom for the standard uncertainties of all the input quantities are infinite, the coverage factor is determined from the normal distribution. Otherwise, the (effective) degrees of freedom for the combined standard uncertainty is estimated from the degrees of freedom for the standard uncertainties associated with the best estimates of the input quantities using the Welch-Satterthwaite formula.

The GUM permits the evaluation of standard uncertainties by any appropriate means. It distinguishes the evaluation by the statistical treatment of repeated observations as a Type A evaluation of uncertainty, and the evaluation by any other means as a Type B evaluation of uncertainty. In evaluating the combined standard uncertainty, both types of evaluation are to be characterized by variances (squared standard uncertainties) and treated in the same way.

Full details of this procedure and the additional assumptions on which it is based are given in the GUM.

The purpose of this Technical Specification is to provide additional detail on the evaluation of uncertainty by statistical means, concentrating on b) above, whether obtained by repeated measurement of the input quantities or of the entire measurement.

ISO/TS 21749:2005

In this Technical Specification the term artefact is often used in the context of measurement. This usage is to be given a general interpretation in that the measurement may also relate to a bulk or chemical item, etc.

4.2 Check standards

A check standard is a standard required to have the following properties.

- It shall be capable of being measured periodically. a)
- It shall be close in material content and geometry to the production items. b)
- It shall be a stable artefact. C)
- d) It shall be available to the measurement process at all times.

Subject to its having these properties, an ideal check standard is an artefact selected at random from the production items, if appropriate, and reserved for this purpose.

Examples of the use of check standards include

- measurements on a stable artefact, and
- differences between values of two reference standards as estimated from a calibration experiment.

Methods for analysing check standard measurements are treated in 5.2.3.

In this Technical Specification, the term "check standard" is to be given a general interpretation. For instance, a bulk or chemical item may be used.

4.3 Steps in uncertainty evaluation

4.3.1 The first step in the uncertainty evaluation is the definition of the measurand for which a measurement result is to be reported for the test item. Special care should be taken to provide an unambiguous definition of the measurand, because the resulting uncertainty will depend on this definition. Possibilities include

- quantity at an instant in time at a point in space,
- quantity at an instant in time averaged over a specified spatial region,
- quantity at a point in space averaged over a time period.

For instance, the measurands corresponding to the hardness of a specimen of a ceramic material are (very) different

- a) at a specified point in the specimen, or
- b) averaged over the specimen.

4.3.2 If the value of the measurand can be measured directly, the evaluation of the standard uncertainty depends on the number of repeated measurements and the environmental and operational conditions over which the repetitions are made. It also depends on other sources of uncertainty that cannot be observed under the conditions selected to repeat the measurements, such as calibration uncertainties for reference standards. On the other hand, if the value of the measurand cannot be measured directly, but is to be calculated from measurements of secondary quantities, the model (or functional relationship) for combining the various quantities must be defined. The standard uncertainties associated with best estimates of the secondary quantities are then needed to evaluate the standard uncertainty associated with the value of the measurand.

The steps to be followed in an uncertainty evaluation are outlined as follows.

https://standards.iteh.ai/catalog/standards/sist/218c8386-79cf-4b05-84a5-

- a) Type A evaluations: 8ff0e60de301/iso-ts-21749-2005
 - 1) If the output quantity is represented by *Y*, and measurements of *Y* can be replicated, use an ANOVA model to provide estimates of the variance components, associated with *Y*, for random effects from
 - replicated results for the test item,
 - measurements on a check standard,
 - measurements made according to a designed experiment.
 - 2) If measurements of *Y* cannot be replicated directly, and the model

 $Y = f(X_1, X_2, ..., X_n)$

is known, and the input quantities X_i can be replicated, evaluate the uncertainties associated with the best estimates x_i of X_i ; then the law of propagation of uncertainty can be used.

- 3) If measurements of *Y* or X_i cannot be replicated, refer to Type B evaluations.
- b) Type B evaluations: evaluate a standard uncertainty associated with the best estimate of each input quantity.
- c) Aggregate the standard uncertainties from the Type A and Type B evaluations to provide a standard uncertainty associated with the measurement result.
- d) Compute an expanded uncertainty.

4.4 Examples in this Technical Specification

The purpose of the examples in various clauses of this Technical Specification and the more detailed case study in Clause 8 is to demonstrate the evaluation of uncertainty associated with measurement processes having several sources of uncertainty. The reader should be able to generalize the principles illustrated in these sections to particular applications. The examples treat the effect of both random effects and systematic effects in the form of bias on the measurement result. There is an emphasis on quantifying uncertainties observed over time, such as those for time intervals defined as short-term (repeatability) and for intermediate measures of precision such as day-to-day or run-to-run, as well as for reproducibility. For the reader's purpose, the time intervals should be defined in a way that makes sense for the measurement process in question.

To illustrate strategies for dealing with several sources of uncertainty, data from the Electronics and Electrical Engineering Laboratory of the National Institute of Standards and Technology (NIST), USA, are featured. The measurements in question are volume resistivities (Ω -cm) of silicon wafers. These data were chosen for illustrative purposes because of the inherent difficulties in measuring resistivity by probing the surface of the wafer and because the measurand is defined by an ASTM test method and cannot be defined independently of the method.

The intent of the experiment is to evaluate the uncertainty associated with the resistivity measurements of silicon wafers at various levels of resistivity (Ω ·cm), which were certified using a four-point probe wired in a specific configuration. The test method is ASTM Method F84. The reported resistivity for each wafer is the average of six short-term repetitions made at the centre of the wafer.

5 Type A evaluation of uncertainty ANDARD PREVIEW

5.1 General

5.1.1 Generally speaking, any observation that can be repeated (see *GUM*, 3.1.4 to 3.1.6) can provide data suitable for a Type A evaluation. Type A evaluations can be based on (for example) the following:

(standards.iteh.ai)

- repeated measurements on the item under test, in the course of, or in addition to, the measurement necessary to provide the result;
- measurements carried out on a suitable test material during the course of method validation, prior to any measurements being carried out;
- measurements on check standards, that is, test items measured repeatedly over a period of time to monitor the stability of the measurement process, where appropriate;
- measurements on certified reference materials or standards;
- repeated observations or determination of influence quantities (for example, regular or random monitoring
 of environmental conditions in the laboratory, or repeated measurements of a quantity used to calculate
 the measurement result).

5.1.2 Type A evaluations can apply both to random and systematic effects (*GUM*, 3.2). The only requirement is that the evaluation of the uncertainty component is based on a statistical analysis of series of observations. The distinction with regard to random and systematic effects is that

- random effects vary between observations and are not to be corrected,
- systematic effects can be regarded as essentially constant over observations in the short term and can, theoretically at least, be corrected or eliminated from the result.

Sometimes it is difficult to distinguish a systematic effect from random effects and it becomes a question of interpretation and the use of related statistical models. In general, it is not possible to separate random and systematic effects.

The *GUM* recommends that generally all systematic effects are corrected and that consequently the only uncertainty from such sources are those of the corrections. The role of time in the evaluation of Type A uncertainty using nested designs is discussed in 5.2. The uncertainties associated with measurement configuration and material inhomogeneity, respectively, are discussed in 5.3 and 5.4. Guidance on how to assess and correct for bias due to measurement configurations and to evaluate the associated uncertainty is given in 5.5. The manner in which the source of uncertainty affects the reported value and the context for the uncertainty determine whether an analysis of a random or systematic effect is appropriate.

Consider a laboratory with several instruments of a certain type, regarded as representative of the set of all instruments of that type. Then the differences among the instruments in this set can be considered to be a random effect if the uncertainty statement is intended to apply to the result of any particular instrument, selected at random, from the set.

Conversely, if the uncertainty statement is intended to apply to one (or several) specific instrument, the systematic effect of this instrument relative to the set is the component of interest.

5.2 Role of time in Type A evaluation of uncertainty

5.2.1 Time-dependent sources of uncertainty and choice of time intervals

Many random effects are time-dependent, often due to environmental changes. Three levels of timedependent fluctuations are discussed and can be characterized as

- a) short-term fluctuations (repeatability or instrument precision),
- b) intermediate fluctuations (day-to-day Sor Toperator Toperator or equipment-to-equipment, known as intermediate precision) and ards. iteh. ai/catalog/standards/sist/218c8386-79cf-4b05-84a5-8ff0e60de301/iso-ts-21749-2005
- c) long-term fluctuations [run-to-run or stability (which may not be a concern for all processes) or intermediate precision].

This characterization is only a guideline. It is necessary for the user to define the time increments that are of importance in the measurement process of concern, whether they are minutes, hours or days.

One reason for this approach is that much modern instrumentation is exceedingly precise (repeatable measurements) in the short term, but changes over time, often caused by environmental effects, can be the dominant source of uncertainty in the measurement process. An uncertainty statement may be inappropriate if it relates to a measurement result that cannot be reproduced over time. A customer is entitled to know the uncertainties associated with the measurement result, regardless of the day or time of year when the measurement was made.

Two levels of time-dependent components are sufficient for describing many measurement processes. Three levels may be needed for new measurement processes or processes whose characteristics are not well understood. A three-level design is considered, with a two-level design as a special case.

Nested designs having more than three levels are not considered in this Technical Specification, but the approaches discussed can be extended to them. See ISO 5725-3.

5.2.2 Experiment using a three-level design

5.2.2.1 A three-level nested design is generally recommended for studying the effect of sources of variability that manifest themselves over time. Data collection and analysis are straightforward, and there is usually no need to estimate interaction terms when dealing with time-dependent effects. Nested designs can