INTERNATIONAL STANDARD

ISO 10576-1

First edition
2003-03-01

Statistical methods — Guidelines for the evaluation of conformity with specified requirements —

Part 1: General principles

Méthodes statistiques — Lignes directrices pour l'évaluation de la conformité à des exigences spécifiques —

Partie 1: Principes généraux

Reference number
ISO 10576-1:2003(E)

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

Page

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.

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 10576-1 was prepared by Technical Committee ISO/TC 69, *Applications of statistical methods*, Subcommittee SC 6, *Measurement methods and results*.

Introduction

Conformity testing is a systematic examination of the extent to which an entity conforms to a specified criterion. The objective is to provide assurance of conformity, either in the form of a supplier's declaration, or of a third party certification (see ISO/IEC Guide 2, 1996). A specification is usually formulated as a single limiting value, LV, or as a set of (upper and lower) limiting values for a measurable characteristic. When the specification refers, e.g. to health-related characteristics, the limiting values are sometimes termed *threshold limit value* TLV, or *permissible exposure limits*, PEL.

Whenever conformity testing involves measurement or sampling uncertainty, it is common practice to invoke elements from the theory of statistical hypothesis testing to provide a formal procedure. With the knowledge of the measurement procedure and of its behaviour with regard to the uncertainty of its outcomes it is possible to estimate and minimize the risk of making erroneous declarations of conformity or non-conformity to the specifications. An operational way of formulating requirements of assurance is to require that whenever an entity has been declared to be conforming, this status should not be altered by subsequent measurements on the entity, even using more precise measurements (e.g. a better measurement method or technology). Or, in terms of risks, the risk of (erroneously) declaring a non-conforming entity to be conforming shall be small. Consequently, it is necessary to tolerate a (large) risk that an entity, which only marginally conforms, will fail to be declared as conforming. Applying a two-stage procedure instead of a one-stage procedure will in general decrease this risk.

When a test for non-conformity is performed, similar considerations are valid.

In this part of ISO 10576, this issue is addressed in respect of the construction of specifications and the testing of output from production or service processes for conformity and non-conformity with specifications.

The problems of how to determine the relevant components of uncertainty and how to estimate them will be addressed in a future ISO 10576-2.

Because of the apparent similarity to acceptance sampling procedures, it is sometimes seen that acceptance sampling plans are used in conformity testing activities. Acceptance sampling and conformity testing activities both utilize elements of hypothesis testing (see e.g. ISO 2854[2]). It is, however, important to realise that the objectives of the two activities are fundamentally different and in particular the two activities imply different approaches to the risk involved (see ISO 2854[2] and Holst[9]).

Statistical methods — Guidelines for the evaluation of conformity with specified requirements —

Part 1: General principles

1 Scope

This part of ISO 10576 sets out guidelines:

a) for drafting requirements that may be formulated as limiting values for a quantifiable characteristic;

b) for checking conformity to such requirements when the test or measurement result is subject to uncertainty.

This part of ISO 10576 is applicable whenever the uncertainty may be quantified according to the principles laid down in GUM. The term uncertainty is thus a descriptor for all elements of variation in the measurement result, including uncertainty due to sampling.

It is outside the scope of this part of ISO 10576 to give rules for how to act when an inconclusive result of a conformity test has been obtained.

NOTE Neither on the nature of the entity subject to the requirements nor on the quantifiable characteristic are there limitations. Examples of entities together with quantifiable characteristics are given in Table A.1.

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 3534-2:1993, *Statistics — Vocabulary and symbols — Part 2: Statistical quality control*

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 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

ISO 5725-4:1994, *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 5725-5:1998, *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 5725-6:1994, *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):*1993[1)], BIPM/IEC/IFCC/ISO/IUPAC/IUPAP/OIML

3 Terms and definitions

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

3.1
limiting values
specification limits
L
specified values of the characteristic giving upper and/or lower bounds of the permissible values

[ISO 3534-2:1993, 1.4.3]

3.2
lower specification limit
L_{SL}
lower bound of the permissible values of the characteristic

3.3
upper specification limit
U_{SL}
upper bound of the permissible values of the characteristic

3.4
conformity test
systematic evaluation by means of testing of the extent to which a product, process or service fulfils specified requirements

3.5
region of permissible values
interval or intervals of all permissible values of the characteristic

NOTE Unless otherwise stated in the specification, the limiting values belong to the region of permissible values.

3.6
region of non-permissible values
interval or intervals of all values of the characteristic that are not permissible

NOTE Figure 1 displays various possibilities for the partitioning of the region of possible values of the characteristic in regions of permissible and non-permissible values.

1) Published in 1993 but corrected and reprinted in 1995.

3.7
uncertainty interval
interval derived from the actual measurement of the characteristic and its uncertainty, covering the values that could reasonably be attributed to this characteristic

NOTE 1 An uncertainty interval may be the symmetric interval around the measurement result as defined in 6.2.1 of GUM:1993.

NOTE 2 When the uncertainty has been obtained only by Type A evaluations of uncertainty components, the uncertainty interval may be in the form of a confidence interval for the value of the characteristic (see e.g., 2.57 of ISO 3534-1:1993 and G.3 of GUM:1993).

3.8
two-sided confidence interval
when T_1 and T_2 are two functions of the observed values such that, θ being a population parameter to be estimated, the probability $\mathrm{P_r}(T_1 \leqslant \theta \leqslant T_2)$ is at least equal to $(1-\alpha)$ [where $(1-\alpha)$ is a fixed number, positive and less than 1], the interval between T_1 and T_2 is a two-sided $(1-\alpha)$ confidence interval for θ

[ISO 3534-1:1993, 2.57]

3.9
confidence coefficient
confidence level
the value $(1-\alpha)$ of the probability associated with a confidence interval or a statistical coverage interval

[ISO 3534-1:1993, 2.59]

4 Specification of requirements

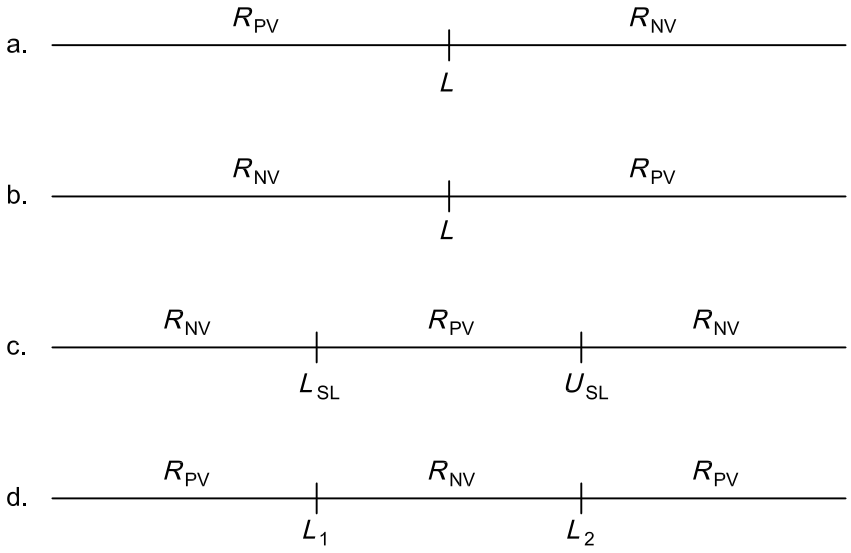
4.1 Requirements for definition of limiting values

4.1.1 The entity shall be clearly and unambiguously specified.

4.1.2 The quantifiable characteristic of the entity shall be clearly and unambiguously specified. The value of the characteristic shall be determined by means of a measurement or test procedure that enables an assessment of the uncertainty of the measurement to be made.

4.1.3 The measurement or test procedure should be a standardized procedure.

4.1.4 The uncertainty of the measurement shall neither explicitly nor implicitly be referred to in the designation of the limiting values.



NOTE R_{PV} denotes Region of permissible values while R_{NV} denotes Region of non-permissible values.

The specification limits are denoted L, L_{SL}, U_{SL}, L_1 and L_2.

Figure 1 — Division of the domain for the characteristic

4.2 Reporting of limiting values

The reporting of limiting values shall be the result of the drafting given in 4.1.1 and 4.1.2.

The range of permissible values of a quantifiable characteristic may be limited to only one side or to both sides. Limits are therefore of two kinds:

— double limits, consisting of an upper *and* a lower limit;

— single limit, i.e. either an upper limit *or* a lower limit.

EXAMPLE 1 Double limits

For a single item in the form of a barrel of motor oil (i.e. the entity) the requirements for the kinematic viscosity of the oil (i.e. the characteristic) could be:

the kinematic viscosity shall be not less than $0{,}5 \times 10^{-5}$ m^2/s and no greater than $1{,}00 \times 10^{-5}$ m^2/s.

EXAMPLE 2 Double limits

For one lot of bottles with frying oil (i.e. the entity) the requirements for the average boiling point at the atmospheric pressure of 101,6 kPa for the oil in the bottles (i.e. the characteristic) could be:

the average boiling point shall be within the interval 105,0 °C to 115,0 °C.

EXAMPLE 3 Single upper limit

For a shipment of crude oil (i.e. the entity) the requirements for the sulfur mass fraction (i.e. the characteristic) in the bulk could be:

the sulfur mass fraction shall be no greater than 2 %.

EXAMPLE 4 Single upper limit

For an individual (i.e. the entity) the requirements for the concentration of lead in blood (i.e. the characteristic) could be:

the concentration of lead shall be no greater than 0,96 µmol/l.

EXAMPLE 5 Single lower limit

For a lot of bitumen (i.e. the entity) the requirements for the solubility of the bitumen in kerosene at 20 °C (i.e. the characteristic) could be:

the solubility of the bitumen in kerosene at 20 °C shall be not less than a mass fraction of 99 %.

EXAMPLE 6 Single upper limit

For a shipment of apples (i.e. the entity) the requirements for mass fraction of the apples infected with pests (i.e. the characteristic) could be:

the mass fraction of apples infected with pests shall be less than 0,2 %.

Due to the variation of the mass of the individual apples, the mass fraction of infected apples will usually be different from the number fraction of infected apples.

NOTE In many cases (e.g. in the environmental field), an additional implied limit such as 0 %, 0,0 kg/l and 100 % can be ignored when considering a single limit because they are theoretical and/or physical limits and therefore need not necessarily to be specified.

5 Uncertainty of results

5.1 General

When comparing a measurement or test result with the limiting values, it is necessary to take into consideration the uncertainty of the measurement result. The uncertainty shall be determined according to the provisions of the GUM. ISO 5725, parts 1 to 6, may also be consulted to help identify some of the components of uncertainty.

NOTE This implies that the contributions to the uncertainty from all stages in the measurement procedure shall be taken into consideration. This also includes any uncertainty due to sampling.

5.2 Reporting the uncertainty of the measurement result

The measurement result of the measured characteristic of interest and the uncertainty of the measurement shall be reported; the uncertainty of the measurement shall be reported as an uncertainty interval. When this interval is a confidence interval, the confidence level $(1 - \alpha)$ shall be reported together with the interval (see 2.57 and 2.59 of ISO 3534-1:1993). Otherwise the coverage factor of the uncertainty interval shall be reported (see 6.2.1 of GUM:1993).

6 Assessing conformity to requirements

6.1 General

A conformity test is a systematic examination (by means of measurement) of whether or not the entity fulfils the specified requirements.

The objective of the conformity test is to provide confidence that the entity fulfils the specified requirements.

This part of ISO 10576 recommends that the conformity test be performed as a two-stage procedure. In the cases where a two-stage procedure either cannot be performed or for other reasons should not be performed, a one-stage procedure is provided.

When a two-stage procedure is performed, there shall be appropriate procedures to evaluate the consistency of the measurement results from the two stages.

NOTE The advantage of the two-stage procedure over the one-stage procedure is the considerably higher probability of declaring conformity for entities with permissible values of the quantity of interest, which are close to the limiting value(s). The disadvantage is a slightly higher probability of declaring conformity for entities with non-permissible values of the quantity of interest which are close to the limiting values. If this increased probability in declaring conformity for non-conforming entities cannot be accepted, a one-stage procedure should be provided.

6.2 The two-stage conformity test

6.2.1 Stage 1

Perform the measurement procedure and calculate the uncertainty of the measurement result.

Conformity to the requirements may be assured if, and only if, the uncertainty interval of the measurement result is inside the region of permissible values.

The second stage of the test shall be performed if, and only if, the uncertainty interval calculated after the first stage includes a specification limit.

6.2.2 Stage 2

Perform the measurement procedure once more and determine an appropriate combination of the two measurement results to form the final measurement result together with the uncertainty of that result.

Conformity to the requirements may be assured if, and only if, the uncertainty interval of the final measurement result is inside the region of permissible values.

If conformity may be assured, either after the first or after the second stage, the statement given in 7.2 may be asserted.

NOTE 1 The uncertainty interval is also considered to be inside the region of permissible values when one of the limits of the uncertainty interval coincides with a limiting value of the specification.

If the uncertainty interval of the measurement result is entirely included in the region of non-permissible values, either after the first or after the second stage, then non-conformity with the requirements may be assured and the statement in 7.3 can be asserted.

NOTE 2 The uncertainty interval is also considered to be inside the region of non-permissible values when one of the limits of the uncertainty interval coincides with a limiting value of the specification.

When the uncertainty interval determined after stage 2 includes a specification limit, the result of the conformity test is inconclusive, and the statement given in 7.4 may be asserted.

NOTE 3 The measurement procedures used in the two stages need not be identical. The appropriate combination of the results from the first and the second stage referred to in stage 2 above also includes situations where e.g., only the result from stage 2 is used as the final measurement result.

Figure 2 displays a flow diagram for the two-stage conformity test.

6.3 The one-stage conformity test

Perform the measurement procedure and calculate the uncertainty of the measurement result.

Conformity to the requirements may be assured if, and only if, the uncertainty interval of the measurement result is inside the region of permissible values.

NOTE 1 The uncertainty interval is also considered to be inside the region of permissible values when one of the limits of the uncertainty interval coincides with a limiting value of the specification.

If the uncertainty interval of the measurement result is entirely included in the region of non-permissible values, then non-conformity with the requirements can be declared and the statement in 7.3 may be asserted.

NOTE 2 The uncertainty interval is also considered to be inside the region of non-permissible values when one of the limits of the uncertainty interval coincides with a limiting value of the specification.

When the uncertainty interval includes a specification limit, the result of the conformity test is inconclusive, and the statement given in 7.4 may be asserted.

6.4 The uncertainty interval given in the form of a confidence interval

The provisions in this subclause refer to situations where the uncertainty interval is given in the form of a confidence interval with confidence level $(1-\alpha)$ (see 5.2). When the specification is given in terms of a single specification limit (case a. or case b. in Figure 1), the probability of an erroneous declaration of conformity is at most $\alpha/2$ for the one-stage procedure and at most $\alpha + \alpha^2/2$ for the two-stage procedure. In the case with two specification limits (case c. or d. in Figure 1), the probability of an erroneous declaration of conformity depends on the average length of the confidence interval. However, when the average length is only a small fraction of the difference between the specification limits, the above expression for the probability of an erroneous declaration of conformity may still be used.

When the uncertainty of the measurements can be assumed to be completely known (i.e. the uncertainty is not calculated from the observations), the probability of declaring conformity with the requirements can be calculated together with the probability of obtaining an inconclusive result from the conformity test.

NOTE Examples will be provided in a future ISO 10576-2.

6.5 Inconclusive result of the conformity test

Especially when the value of the characteristic is in the neighbourhood of a specification limit, there is a large probability that the result of the conformity test will be inconclusive. This is in principle unsatisfactory but is inevitable if a declaration of conformity with the requirements should justify the assertion of the statement in 7.2.

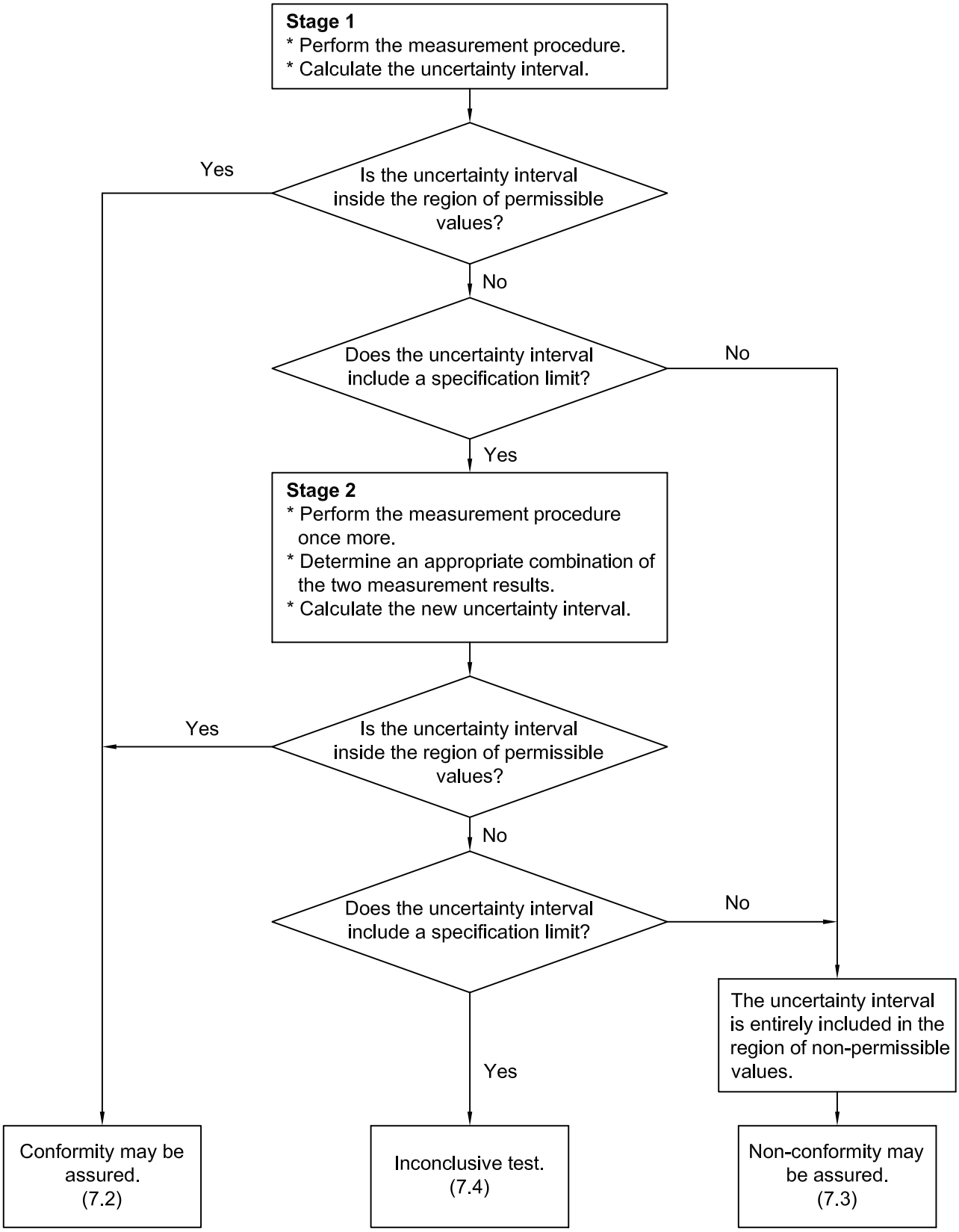


Figure 2 — Flow diagram for the two-stage procedure

7 Reporting the result of the conformity assessment

7.1 General

Due to the variability in measurement, an assertion based upon the measurements may be wrong. The design of the measurement procedure and the test procedure shall therefore take this into account in the reporting of a conformity test.

When reporting the result of a conformity test, the qualitative expressions for assurance of conformity, non-conformity or an inconclusive test given in 7.2, 7.3 and 7.4 shall be supplemented with all the evidence which supports the qualitative expression used.

7.2 Assurance of conformity

Whenever the uncertainty interval of the measurement result is inside the region of permissible values (see 6.1 and 6.2), conformity may be assured.

The assurance of conformity shall have the following wording:

The conformity test has demonstrated beyond any reasonable doubt that the value of the characteristic is in conformity with the requirements.

7.3 Assurance of non-conformity

Whenever the uncertainty interval of the measurement result is inside the region of non-permissible values (see 6.1 and 6.2), non-conformity may be assured.

The assurance of non-conformity shall have the following wording:

The conformity test has demonstrated beyond any reasonable doubt that the value of the characteristic is not in conformity with the requirements.

7.4 Inconclusive result

Whenever neither conformity nor non-conformity with the requirements can be assured in accordance with 6.1 or 6.2, the result of the conformity test is inconclusive.

The report of an inconclusive test result shall have the following wording:

The conformity test has not been able to demonstrate beyond any reasonable doubt that the value of the characteristic is or is not in conformity with the requirements.

Annex A
(informative)

Examples of entities and quantifiable characteristics

Table A.1 — Examples of entities together with quantifiable characteristics

Entity	Quantifiable characteristic of entity			
	Item characteristic	Average	Homogeneity	Relative frequency
Distinguishable item or individual	×	—	×	—
{weight for a balance}	{mass}	—	—	—
Group of distinguishable items (batch or population)	—	×	×	×
{lot of bags of sugar}	—	{the average mass per bag}	{the standard deviation of the mass of bags}	{the percentage of bags with conforming masses}
Process	—	×	×	×
{production of bottles}	—	{the average volume per bottle produced}	{the standard deviation of the volume of the bottles produced}	{the percentage of produced bottles with conforming volumes}
Lot of bulk material (particulate material, liquid or gas)	—	×	×	×
{lot of dolomite}		{the mass fraction of asbestos fibres}	{the standard deviation of the mass fraction of asbestos between specified sampling units}	{the mass fraction of asbestos fibres with conforming length}
Service	—	×	×	×
{treatment of a specific disease}	—	{the average waiting time from the reporting of the disease until the start of the treatment}	{the standard deviation of the waiting time from the reporting of the disease until the start of the treatment}	{the percentage of waiting times for the start of the treatment with conforming length}

The symbol "×" in the cell indicates that the characteristic may be considered for the entity in question. Specific examples are given in accolades { }.

The contents of this table should not be considered exhaustive.

Annex B
(informative)

Examples

B.1 General

The following examples cover only some of the combinations of the entities and quantifiable characteristics given in Table A.1. The examples do not represent any specific important combinations of entity and characteristic of interest.

B.2 Example 1

In a series of fine turned steel shafts, nominal dimensions ∅ 25 mm × 150 mm, the specification limits for the diameter (two point diameter) of each shaft is L_{SL} = 24,9 mm and U_{SL} = 25,0 mm. The entity is thus a shaft and the characteristic is the shaft diameter.

The measurements are performed using an analogue external micrometer with flat measuring anvils, a measuring range of 0 to 25 mm with a Vernier scale interval of 10^{-3} mm. The standard uncertainty of measurement, $u_c = 3{,}79 \times 10^{-3}$ mm, is calculated from a number of contributors (see A.2 of ISO/TS 14253-2:1999). For economic reasons a one-stage conformity test was performed for each of the shafts in the series instead of a two-stage test. The uncertainty intervals were calculated in accordance with 6.2.1 of GUM:1993, using the coverage factor k = 2. The uncertainty intervals around the measurements of three shafts were (24,857 ± 0,007 6) mm; (24,907 ± 0,007 6) mm and (24,962 ± 0,007 6) mm. In accordance with 6.3, the first shaft is declared to be non-conforming and the third shaft is declared to be conforming to the requirements while the conformity test of the second shaft has given an inconclusive result.

B.3 Example 2

According to a list of limiting values, the concentration of lead in blood for individuals shall not exceed 0,97 μmol/l. The entity is thus the blood of an individual. The characteristic is per definition the concentration of trace metal in the blood at the time the blood sample is taken. When a two-stage procedure is used the blood sample is divided into two subsamples and the second sample is only measured if the uncertainty interval after the first stage contains a limiting value (see 6.2). The measurements are performed with a standard measurement procedure which operates with an uncertainty of σ_Y = 0,048 μmol/l[7,8]. The uncertainty interval of a measurement can be expressed in the form of a $(1 - \alpha)$ confidence interval for the value of the characteristic[10,11]. When n independent measurements each with the uncertainty σ_Y are performed and the arithmetic mean of the measurements is Y_1 then the confidence interval is given as

$$Y_1 \pm \frac{u_{1-\alpha/2}\sigma_Y}{\sqrt{n}}$$

where $u_{1-\alpha/2}$ is the $1 - \alpha/2$ quantile of the standard normal distribution[1].

The concentration of Pb in the blood for a particular individual is measured. The individual is only exposed to lead through daily food intake and the exhaust emissions from motor vehicles. The estimate of the Pb concentration from the measurement of the first subsample (n = 1) of blood is calculated as Y_1 0,60 μmol/l. The uncertainty interval given in the form of a 0,95 confidence interval for w is 0,504 μmol/l to 0,693 μmol/l. Since this interval is entirely included in the region of conformity, then, in accordance with 6.3, conformity with the requirements is declared.

The Pb concentration for another individual with a supplementary exposure to lead coming from his daily work is also measured. The measurement result from the first subsample (n = 1) is Y_1 = 1,06 μmol/l and the corresponding 0,95 confidence interval for the Pb concentration is 0,96 μmol/l to 1,15 μmol/l. Since this interval includes the limiting value, the second subsample is measured (n = 1). This measurement result is 1,00 μmol/l. The measurements from the two stages are combined to Y_* = (1,06 + 1,00)/2 μmol/l = 1,03 μmol/l. The confidence interval for the Pb concentration based on the arithmetic mean of the two estimates is calculated from the formula given above (n = 2) resulting in the interval 0,96 μmol/l to 1,10 μmol/l. The limiting value is inside this interval. Thus, it cannot be concluded that the concentration of Pb is in conformity with the requirements. Correspondingly, it cannot be concluded that the Pb concentration is not in conformity with the requirements. In accordance with 6.3, the result from the two conformity tests is therefore inconclusive.

It should be emphasised that the procedure of performing a conformity test for the concentration of lead in human blood given above is not equivalent to the standard procedure currently used.

B.4 Example 3

In a location, it is specified that the total mass of cadmium (Cd) in the discharge water from a power station shall not exceed a daily mass of 5 g in more than 20 % of the days of the measurement period. The entity is thus the process of daily discharges of water from the power station. The characteristic of interest is the 80 % percentile (i.e. the 0,8 quantile) in the distribution of the daily outlet of Cd. The upper specification limit for the percentile is 5 g Cd. Studies of the daily Cd amount in the process of discharge water have indicated that the distribution of the Cd amount can be described by a lognormal distribution. The upper confidence limit, U_{CL}, in a one sided (1 − α) confidence interval for the p quantile in a lognormal distribution based on a sample of n independent measurements is

$$U_{\mathrm{CL}} = \exp\left\{ \bar{X} + \frac{s_x t\left[\delta,(n-1)\right]_{1-\alpha}}{\sqrt{n}} \right\}$$

where

$\bar{X}$ is the arithmetic mean of the logarithm of the n observations;

s_x is the corresponding sample standard deviation;

$t[\delta, (n-1)]_\alpha$ is the α quantile of the non-central t-distribution with (n − 1) degrees of freedom and the non-centrality parameter δ.

With u_p denoting the p quantile of the standard normal distribution, δ is given by $\delta = -u_p\sqrt{n}$. A one-stage conformity test is performed on 10 daily samples of discharge water; each sampled consecutively with an interval of 14 d. The Cd content in each sample is measured, and the daily outlet of Cd is estimated assuming homogeneity of the Cd content in the discharge water. The uncertainty of the individual measurement results (i.e. of the daily outlet of Cd) is negligible compared to the variation in the Cd outlet between the individual days.

The following 10 observations of the daily Cd discharge were obtained (given in grams):

0,348 6; 0,140 8; 0,089 0; 1,141 7; 0,752 4; 0,626 2; 3,756 0; 0,552 0; 0,230 4; 1,722 6

The arithmetic mean and the standard deviation of the natural logarithm of the observations are

$\bar{X}$ = –0,624 837 and s_x= 1,143 79

Since p = 0,80, we have u_{p} = 0,841 621 and thus δ = – 2,661 44. For a 95 % confidence interval (i.e. α = 0,05) for the 80 % percentile, we have

$t[\delta, (n-1)]_{\alpha-1} = t(2{,}661\ 44;9)_{0.95}$ = 5,386 87

The upper limit of the one-sided 95 % confidence interval for the 80 % percentile in the distribution of the daily Cd amount in the discharge water is therefore

$$U_{CL} = \exp(-0{,}624\ 837 + 1{,}143\ 79 \times 5{,}386\ 87/\sqrt{10}) = \exp(1{,}323\ 58) = 3{,}756\ 86$$

As $U_{CL} < 5$, conformity with the requirements may be declared.

B.5 Example 4

Scandinavian dolomite normally contains a minor fraction of asbestos fibres that may damage the health of people handling the dolomite. Therefore, for health reasons, an upper limit has been specified for the mass fraction of asbestos in Scandinavian dolomite used in industry. The upper specification limit is a mass fraction of 0,001 % or 0,1 %. Before lots of dolomite are released for processing they are subjected to a test of conformity to this specification.

The entity is thus a lot of dolomite, and the characteristic of interest is the mass fraction, w, of asbestos fibres in the lot, i.e. the specification is $w \leqslant 0{,}1$ %.

To estimate the mass fraction, a number of primary increments is selected from the lot. From each of the increments, a specified number of laboratory samples is formed and used for analysis. For each primary increment, the average, $\bar{X}$, of the mass fraction found in the corresponding laboratory samples is calculated. When the number of laboratory samples is large, it is known that the distribution (over primary increments) of these averages may be well approximated by a normal distribution with mean w and variance σ^2. The variance σ^2 contains contributions from the variation between primary increments, the variation within primary increments and the measurement uncertainty associated with the analysis of the laboratory samples.

When n primary increments are analysed and the same number of laboratory samples are formed from each of the n primary increments, the mass fraction, w of asbestos fibres in the lot is estimated by the arithmetic mean, $\bar{X}$, of the results from the primary increments i.e.

$$\bar{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

Assuming independent observations are all with the same variance, the variance σ^2 is estimated by the empirical variance:

$$s^2 = \frac{\sum_{i=1}^{n} (X_i - \bar{X})^2}{n-1}$$

A $(1 - \alpha)$ confidence interval for w is given as

$$\bar{X} \pm \frac{t(n-1)_{1-\alpha/2}\, s}{\sqrt{n}}$$

where $t(n-1)_{1-\alpha/2}$ is the $1 - \alpha/2$ quantile of the t-distribution with $n-1$ degrees of freedom[1].

A lot of Scandinavian dolomite was presented for testing conformity to the specification for asbestos content. Since the measurement procedure is very time-consuming, it was decided to perform the conformity test as a two-stage procedure with five primary increments in the first stage and four primary increments in the second stage. From each primary increment 10 laboratory samples were formed for analysis.

In the first stage the following results were obtained (given as mass fractions of asbestos):

0,152 %; 0,070 4 %; 0,077 2 %; 0,073 1 %; 0,055 1 %

Based on the observations from the first stage and using α = 0,05, the following confidence interval for w was obtained: 0,085 6 % $\pm$ (2,776 $\times$ 0,038 1 %)/$\sqrt{5}$ = (0,038 %; 0,133 %). Since the specification limit, 0,1 %, is contained in the interval, it was decided to proceed to the second stage of the conformity test and analyse 10 laboratory samples from each of the remaining four primary increments. The following results were obtained from these samples:

0,082 8 %; 0,067 1 %; 0,074 3 %; 0,056 1 %.

Using the procedure given above the final confidence interval is determined to be

0,078 7 % $\pm$ 2,306 $\times$ 0,029 0/$\sqrt{9}$ % = (0,056 %; 0,101 %).

Since this interval also contains the specification limit, conformity to the requirements has not been demonstrated.

Bibliography

[1] ISO 2602:1980, *Statistical interpretation of test results — Estimation of the mean — Confidence interval*

[2] ISO 2854:1976, *Statistical interpretation of data — Techniques of estimation and tests relating to means and variances*

[3] ISO 9000: 2000, *Quality management systems — Fundamentals and vocabulary*

[4] ISO/TS 14253-2:1999, *Geometrical Product Specifications (GPS) — Inspection by measurement of workpieces and measuring equipment — Part 2: Guide to the estimation of uncertainty in GPS measurement, in calibration of measuring equipment and in product verification*

[5] ISO Guide 2: 1996, *Standardization and related activities — General vocabulary*

[6] *International vocabulary of basic and general terms in metrology*, 1993, BIPM/IEC/IFCC/ISO/IUPAC/ IUPAP/OIML

[7] CHRISTENSEN, J.M., POULSEN, O.M. and ANGLOV, T., Protocol for the design and interpretation of method evaluation in AAS analysis. Application to the determination of lead and manganese in blood. *Journal of Analytical Atomic Spectroscopy,* 1992, vol. 7*,* pp. 329-334

[8] CHRISTENSEN, J.M., Human Exposure to Toxic Metals. Factors influencing Interpretation of Biomonitoring Results, *Science of the Total Environment,* 1995, vol. 166*,* pp. 89-135

[9] HOLST, E., THYREGOD, P. and WILRICH, P.-TH., On conformity testing and the use of two-stage procedures, *International Statistical Review*, 2001, vol. 69 (3)

[10] KRISTIANSEN, J., CHRISTENSEN, J.M. and NIELSEN, J.L., Uncertainty of atomic absorption spectrometry: Applications to the determination of lead in blood. *Mikrochimica Acta*, 1996, vol. 123*,* pp. 241-249

[11] KRISTIANSEN, J. and CHRISTENSEN, J.M., Traceability and uncertainty in analytical measurements. *Annals of Clinical Biochemistry*, 1998, vol. 35, pp. 371-379

ICS 03.120.30

Price based on 15 pages