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Introduction

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Template for comments and secretariat observations

Date: xx/xx/20xx	Document: ISO/DIS xxxx
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1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/ Figure/Table/Note	Type of comment	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	



DRAFT International Standard

ISO/DIS 11352

Water quality — Estimation of measurement uncertainty based on validation and quality control data

*Qualité de l'eau — Estimation de l'incertitude de mesure basée
sur des données de validation et de contrôle qualité*

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ISO/TC 147/SC 2

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2024-09-23

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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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 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

This second edition cancels and replaces the first edition (ISO 11352:2012), which has been technically revised.

The main changes are as follows:

- requirements for replicate measurements for precision estimation have been changed to at least 20 replicates,
- a new informative annex has been introduced to describe how to divide the measurement range into two parts for constant absolute and constant relative uncertainties,
- in [Annex B](#) the calculation of the standard deviation from replicate measurements has been changed to the use of pooled standard deviations,
- the examples in [Annex C](#) have been adjusted,
- the standard has been editorially revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The basic principles of the estimation of measurement uncertainty are set out in ISO/IEC Guide 98-3. There are several ways of estimating measurement uncertainty depending on the purpose of the estimation and the available data; Eurolab TR 1/2007^[1] gives an overview of the main approaches.

This International Standard specifies a set of procedures to enable laboratories to estimate the measurement uncertainty of their results, using an approach based on quality control results and validation data. It is structured in a way that is applicable to analysts that do not have a thorough understanding of metrology or statistics.

Nordtest TR 537^[2] has been used as a basis for developing this International Standard. The approach taken is “top-down”, contrary to the mainly “bottom-up” strategy adopted in ISO/IEC Guide 98-3.

It is statistically acceptable to combine a precision estimate and the uncertainty associated with the bias into one uncertainty measure. The sources of data for this approach are method validation and analytical quality control. The experimental approach specified in this International Standard enables a good coverage of the sources of variation observed during routine use of the analytical method.

Water quality — Estimation of measurement uncertainty based on validation and quality control data

1 Scope

This document specifies approaches for the estimation of measurement uncertainty of chemical and physicochemical methods in single laboratories based on validation data and quality control results obtained within the field of water analysis.

NOTE 1 The principles of the estimation of uncertainty specified in this International Standard are consistent with the principles described in ISO/IEC Guide 98-3.

In this document, the quantification of measurement uncertainty relies on performance characteristics of a measurement procedure obtained from validation and the results of internal and external quality control.

NOTE 2 The approaches specified in this International Standard are mainly based on Nordtest TR 537,^[2] but also QUAM,^[3] and Eurolab TR 1/2007^[1].

NOTE 3 This document only addresses the evaluation of measurement uncertainty for results obtained from quantitative measurement procedures. The uncertainties associated with results obtained from qualitative procedures are not considered.

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/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/IEC Guide 99^[2] and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

trueness

closeness of agreement between the average of an infinite number of replicate measured quantity values and a reference quantity value

[SOURCE: ISO/IEC Guide 99:2007,^[4] 2.14]

3.2

precision

measurement precision

closeness of agreement between indications or measured quantity values obtained by replicate measurements on the same or similar objects under specified conditions

[SOURCE: ISO/IEC Guide 99:2007^[4] 2.15]

3.3

error

measurement error

measured quantity value minus a reference quantity value

[SOURCE: ISO/IEC Guide 99:2007^[4] 2.16]

3.4

systematic error

systematic measurement error

component of measurement error that in replicate measurements remains constant or varies in a predictable manner

[SOURCE: ISO/IEC Guide 99:2007^[4] 2.17]

3.5

bias

measurement bias

estimate of a systematic measurement error

[SOURCE: ISO/IEC Guide 99:2007^[4] 2.18]

3.6

random error

random measurement error

component of measurement error that in replicate measurements varies in an unpredictable manner

[SOURCE: ISO/IEC Guide 99:2007^[4] 2.19]

3.7

repeatability conditions

condition of measurement, out of a set of conditions that includes the same measurement procedure, same operators, same measuring system, same operating conditions and same location, and replicate measurements on the same or similar objects over a short period of time

[SOURCE: ISO/IEC Guide 99:2007^[4] 2.20]

3.8

repeatability

measurement precision under a set of repeatability conditions of measurement

[SOURCE: ISO/IEC Guide 99:2007^[4] 2.20]

3.9

analytical run

series of measurements made under repeatability conditions

3.10

intermediate precision conditions

condition of measurement, out of a set of conditions that includes the same measurement procedure, same location, and replicate measurements on the same or similar objects over an extended period of time, but may include other conditions involving changes

Note 1 to entry: The changes can include new calibrations, calibrators, operators, and measuring systems.

[SOURCE: ISO/IEC Guide 99:2007,^[4] 2.22]

3.11

intermediate precision

measurement precision under a set of intermediate precision conditions of measurement

[SOURCE: ISO/IEC Guide 99:2007,^[4] 2.23]

3.12

within-laboratory reproducibility

intermediate measurement precision where all variations that may occur within one laboratory are included

3.13

reproducibility conditions

condition of measurement, out of a set of conditions that includes different locations, operators, measuring systems, and replicate measurements on the same or similar objects

[SOURCE: ISO/IEC Guide 99:2007,^[4] 2.24]

3.14

reproducibility

measurement precision under reproducibility conditions of measurement

[SOURCE: ISO/IEC Guide 99:2007,^[4] 2.25]

3.15

uncertainty

measurement uncertainty

non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

[SOURCE: ISO/IEC Guide 99:2007,^[4] 2.26]

3.16

standard uncertainty

standard measurement uncertainty

measurement uncertainty expressed as a standard deviation

[SOURCE: ISO/IEC Guide 99:2007,^[4] 2.30]

3.17

combined standard uncertainty

combined standard measurement uncertainty

standard measurement uncertainty that is obtained using the individual standard measurement uncertainties associated with the input quantities in a measurement model

[SOURCE: ISO/IEC Guide 99:2007,^[4] 2.31]

3.18

relative standard measurement uncertainty

standard measurement uncertainty divided by the absolute value of the measured quantity value

[SOURCE: ISO/IEC Guide 99:2007,^[4] 2.32]

3.19

target measurement uncertainty

target uncertainty

measurement uncertainty specified as an upper limit and decided on the basis of the intended use of measurement results

[SOURCE: ISO/IEC Guide 99:2007,^[4] 2.34]

3.20**expanded uncertainty****expanded measurement uncertainty**

product of a combined standard measurement uncertainty and a factor larger than the number one

Note 1 to entry: The term “factor” in this definition refers to a coverage factor.

[SOURCE: ISO/IEC Guide 99:2007,^[4] 2.35]

3.21**coverage factor**

number larger than one by which a combined standard measurement uncertainty is multiplied to obtain an expanded measurement uncertainty

[SOURCE: ISO/IEC Guide 99:2007,^[4] 2.38]

4 Symbols

b	bias estimated as the difference between mean measured value and an accepted reference value
b_i	bias of the i th reference material respectively deviation from the complete recovery (100 %) of the i th recovery experiment
b_{rms}	root mean square (quadratic mean) of individual bias values respectively of the deviations from recovery experiments
c	concentration
c_T	transition point between absolute and relative uncertainties
D_i	difference between the measurement result and the assigned value of the i th sample of the inter-laboratory comparison
D_{rms}	root mean square (quadratic mean) of the differences
i	variable related to an observation of a series
j	variable related to a source of uncertainty
J	total number of sources of uncertainty
k	coverage factor
n_{ilc}	number of analysed interlaboratory comparison samples
n_M	number of measurements
$n_{p,i}$	number of participating laboratories for sample i
n_r	number of reference materials
n_η	number of recovery experiments
s	standard deviation
s_b	standard deviation of the measured values of the reference material
s_p	pooled standard deviation
$s_{R,i}$	reproducibility standard deviation from the interlaboratory comparison for sample i

s_{Rw}	within-laboratory reproducibility standard deviation
$s_{Rw,rel}$	Relative within-laboratory reproducibility standard deviation
U	expanded uncertainty
U_{rel}	relative expanded uncertainty
u_c	combined standard uncertainty
$u_{c,rel}$	combined relative standard uncertainty
u_j	standard uncertainties from different sources j
$u_{j,rel}$	relative standard uncertainties from different sources j
u_{add}	standard uncertainty in the concentration of the analyte added
u_b	standard uncertainty component associated with bias
u_{conc}	standard uncertainty of the concentration of the addition solution
$\bar{u}_{C_{ref}}$	mean standard uncertainty of the reference values or mean standard uncertainty of the assigned values of the interlaboratory comparison samples
$u_{C_{ref}}$	standard uncertainty of the reference value
$u_{C_{ref},i}$	standard uncertainty of the assigned value of the interlaboratory sample i
u_{Rw}	standard uncertainty component for the within-laboratory reproducibility
$u_{Rw,rel}$	Relative standard uncertainty component for the within-laboratory reproducibility
$u_{r,range}$	standard uncertainty component from the range control chart (obtained under repeatability conditions)
$u_{Rw,run}$	standard uncertainty component resulting from variations between analytical runs
$u_{Rw,stand}$	standard uncertainty component of the results from the standard solution which is used as quality control sample
u_V	standard uncertainty component of the volume added
$u_{V,b}$	systematic standard uncertainty component of the volume added
$u_{V,rep}$	random standard uncertainty component of the volume added (obtained under repeatability conditions)
$\varepsilon_{V,max}$	maximum deviation of the volume from the specified value (producer information)
η	recovery

5 Principle

A measurement result of a laboratory is an estimate of the value of the measurand. The quality of this estimate depends on the uncertainty that is inherent to the measurement result. In principle, the measurement uncertainty is a property of individual measurement results. The estimation of the measurement uncertainty for each individual measurement result is usually not necessary, if the laboratory can demonstrate that the identified critical influencing factors are under control.^[5] In this International Standard, the measurement uncertainty is, therefore, determined for a set of similar measurement results. Generally, it is assumed that the set of measurement results obtained with a specific analytical method is obtained under controlled

conditions. The estimation of the measurement uncertainty applies to all of the measurement results within the set, independently of, for example, sample matrix variations or different analysts.

This International Standard specifies procedures for the estimation of measurement uncertainty within the scope of the analytical method, and generally, random and systematic errors need to be considered. The estimation of measurement uncertainty is based on analytical quality control results and validation data which represent the within-laboratory reproducibility, and the bias.

6 Procedure

The procedure for the estimation of measurement uncertainty consists of the steps shown schematically in [Figure 1](#). It gives references to appropriate clauses and sub-clauses within this International Standard.

In general, the bias (systematic error) and the within-laboratory reproducibility (random error) are determined independently using suitable data from method validation and analytical quality control results.

The combined measurement uncertainty, i.e. the root of the quadratic sum of the uncertainty component for the within-laboratory reproducibility and the uncertainty component associated with bias, is multiplied by a factor of 2 to obtain the expanded uncertainty at a confidence level of approximately 95 %.

If the measurement uncertainty varies significantly, depending on the matrix and/or concentration range, the uncertainty estimation shall be made separately for each matrix and/or concentration range.

The generic term 'concentration' is used on its own, i.e. unqualified, when a generality is required. It represents the family of terms which includes, mass fraction, mass concentration, amount of substance concentration etc.

7 Preparative considerations for the estimation of measurement uncertainty

7.1 Specification of the measurement

Before starting the estimation of measurement uncertainty, it is necessary that the analyst specifies the analytical method under consideration, and the objectives and purposes of the measurement. The following list is a minimal checklist for this specification.

The specification comprises:

- the measurand;
- the measurement procedure;
- the field of application (matrices, concentration range).

7.2 Specification of the parametric form in which the measurement uncertainty is reported

The expanded uncertainty, U , is reported either as an absolute uncertainty value or as a relative uncertainty value. For results near the limit of quantification, the uncertainty is often found to be constant and can therefore be expressed as an absolute value. When results are well above the limit of quantification, the uncertainty is often proportional to the analyte concentration and can therefore be expressed as a relative value.

EXAMPLE Determination of a heavy metal (limit of quantification: 5 µg/l).

for concentration range 5 µg/l to 20 µg/l $U = 1 \text{ µg/l}$

for concentration >20 µg/l $U_{\text{rel}} = 5 \%$

More information including different approaches to estimate the transition point between absolute and relative uncertainties is given in [Annex A](#).