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Air quality — Guidelines for estimating measurement uncertainty

Qualité de l'air — Lignes directrices pour estimer l'incertitude de mesure

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Foreword

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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.

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Introduction

The general concept of uncertainty estimation is described in the *Guide to the Expression of Uncertainty in Measurement* (GUM). Practical considerations of the GUM are focussed on evaluation of series of unbiased observations. In air quality measurements, series of observations may rarely be considered unbiased due to the presence of random effects not varying throughout a series of observations.

This International Standard supports evaluation of random effects causing variation or bias in series of observations for the purpose of uncertainty estimation. Appropriate data may be collected in experimental designs providing comparison with reference material, or with reference instruments, or with independent measurements of the same type. In provision of experimental data for uncertainty estimation, it is important to ensure representativeness for variations and bias occurring in intended use of the method of measurement.

Generic guidance and statistical procedures presented by this International Standard are addressed to technical experts of air quality measurement, acting, e.g. in standardization, validation or documentation of methods of measurement in ambient air, indoor air, stationary source emissions, workplace atmospheres or meteorology.

This International Standard does not provide comprehensive information on planning and execution of experimental designs to be evaluated for the purpose of uncertainty estimation.

Uncertainties of results of measurement caused by incomplete time-coverage of measurement data are not considered in this document, but in ISO 11222[2]. Uncertainties of results of measurement induced by incomplete spatial coverage by measurement data are not considered in this document.

Air quality — Guidelines for estimating measurement uncertainty

1 Scope

This International Standard provides comprehensive guidance and specific statistical procedures for uncertainty estimation in air quality measurements including measurements of ambient air, stationary source emissions, indoor air, workplace atmospheres and meteorology. It applies the general recommendations of the *Guide to the Expression of Uncertainty in Measurement* (GUM) to boundary conditions met in air quality measurement. The boundary conditions considered include measurands varying rapidly in time, as well as the presence of bias in a series of observations obtained under conditions of intended use of methods of air quality measurement.

The methods of measurement considered comprise

— methods corrected for systematic effects by repeated observation of reference materials,

— methods calibrated by paired measurement with a reference method,

— methods not corrected for systematic effects because they are unbiased by design, and

— methods not corrected for systematic effects in intended use deliberately taking into account a bias.

Experimental data for uncertainty estimation can be provided either by a single experimental design in a direct approach or by a combination of different experimental designs in an indirect approach.

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/IEC Guide 98:1995, *Guide to the expression of uncertainty in measurement (GUM)*

3 Terms and definitions

3.1
uncertainty (of measurement)
measurement uncertainty
parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand

[ISO/IEC Guide 98:1995, B.2.18; VIM:1993, 3.9]

3.2
standard uncertainty
uncertainty of the result of measurement expressed as a standard deviation

[ISO/IEC Guide 98:1995, 2.3.1]

NOTE The standard uncertainty of a result of measurement is an estimate of the standard deviation of the population of all possible results of measurement which can be obtained by means of the same method of measurement for the measurand exhibiting a unique value.

3.3
combined standard uncertainty
standard uncertainty of the result of measurement when that result is obtained from the values of a number of other input quantities, equal to the positive square root of a sum of terms, the terms being the variances or covariance of these other quantities weighted according to how the measurement result varies with changes in these quantities

[ISO/IEC Guide 98:1995, 2.3.4]

NOTE The adjective “combined” can be omitted often without loss of generality.

3.4
expanded uncertainty
quantity defining an interval $[y - U_p(y); y + U_p(y)]$ about the result of a measurement y that may be expected to encompass a large fraction p of the distribution of values that could reasonably be attributed to the measurand

NOTE 1 Adapted from ISO/IEC Guide 98:1995, 2.3.5.

NOTE 2 If the uncertainty has been obtained mainly by Type A evaluation, the interval $[y - U_p(y); y + U_p(y)]$ can be understood as confidence interval for the true value of the measurand on a level of confidence p.

NOTE 3 The interval $[y - U_p(y); y + U_p(y)]$ characterizes the range of values within which the true value of the measurand is confidently expected to lie (see ISO/IEC Guide 98:1995, 2.2.4).

3.5
coverage factor
numerical factor used as multiplier of the combined standard uncertainty in order to obtain an expanded uncertainty

[ISO/IEC Guide 98:1995, 2.3.6]

3.6
coverage probability
fraction of results of measurement expected to be encompassed by a specified interval

3.7
Type A evaluation (of uncertainty)
method of evaluation of uncertainty by the statistical analysis of series of observations

[ISO/IEC Guide 98:1995, 2.3.2]

3.8
Type B evaluation (of uncertainty)
method of evaluation of uncertainty by means other than the statistical analysis of series of observations

[ISO/IEC Guide 98:1995, 2.3.3]

3.9
standard deviation
positive square root of the variance

[ISO/IEC Guide 98:1995, C.2.12]

NOTE In general, the standard deviation of the population of a random variable X is estimated by the positive square root of an estimate of the variance of the population of X.

3.10
experimental standard deviation
for a series of N measurements of the same measurand, the quantity $s(x)$ characterizing the dispersion of the results is given by the formula

$$s(x) = \sqrt{\sum_{j=1}^{N} \frac{\left(x(j) - \bar{x}\right)^2}{N-1}}$$

$x(j)$ being the result of the jth measurement and $\bar{x}$ being the arithmetic mean of the N results considered

NOTE 1 Adapted from ISO/IEC Guide 98:1995, B.2.17.

NOTE 2 $s^2(x)$ is an unbiased estimate of the variance $\sigma^2(X)$ of the investigated random variable X, if the series of observations $x(j)$ with j = 1 to N is unbiased.

3.11
variance
the expectation of the square of the centred random variable:

$$\sigma^2(X) = E\left\{\left[X - E(X)\right]^2\right\}$$

[ISO/IEC Guide 98:1995, C.2.11]

NOTE The population variance $\sigma^2(X)$ of a random variable X can be estimated by the square of the experimental standard deviation $s^2(x)$ of a simple random sample of unbiased observations $x(j)$ with j = 1 to N of the random variable X. Otherwise, $s^2(x)$ underestimates the population variance.

3.12
covariance
mean of the product of two centred random variables in their joint probability distribution

NOTE 1 Adapted from ISO 3534-1: 2006, 2.43.

NOTE 2 The covariance cov(x, y) is a sample statistic used to estimate the covariance of the populations of x and y.

3.13
expectation
expected value

1) For a discrete random variable X taking the values x_i with probabilities p_i, the expectation, if it exists, is $E(X) = \Sigma\, p_i\, x_i$, the sum being extended over all values x_i which may be taken by X.

2) For a continuous random variable X having the probability density function $f(x)$, the expectation, if it exists, is $E(X) = \int x \cdot f(x) \cdot \mathrm{d}x$, the integral being extended over the interval(s) of variation of X.

[ISO/IEC Guide 98:1995, C.2.9]

3.14
degrees of freedom
in general, the number of terms in a sum minus the number of constraints on the terms of the sum

[ISO/IEC Guide 98:1995, C.2.31]

NOTE For a variance estimate, the (effective) number of degrees of freedom can be understood as the number of independent pieces of information used to obtain that variance estimate.

3.15
measurement
set of operations having the object of determining the value of a quantity

[VIM:1993, 2.1]

3.16
result of measurement
value attributed to the measurand, obtained by measurement

[VIM:1993, 3.1]

3.17
sensitivity coefficient
deviation of the result of measurement divided by the deviation of an influence quantity causing the change, if all other influence quantities are kept constant

3.18
measurand
particular quantity subject to measurement

[VIM:1993, 2.6]

NOTE The measurand is considered to exhibit a unique value at least for the time period needed for a single measurement.

3.19
measuring system
complete set of measuring instruments and other equipment with operating procedures to carry out specified air quality measurements

[ISO 11222:2002, 3.9]

NOTE A measuring system is a technical realization of a method of measurement. Method documentation is considered part of a measuring system.

3.20
reference material
RM
material or substance for which one or more properties are sufficiently homogeneous and well established to be used for the calibration and/or the validation of a measuring system

NOTE 1 Adapted from VIM:1993, 6.13.

NOTE 2 A reference material may be in the form of a pure or mixed gas, liquid or solid.

3.21
systematic effect
Influence causing a bias that is expected to occur consistently in each series of observations obtained in repeated or parallel execution of the measurement

3.22
random effect
influence causing either random variation or a bias of random value (inconsistent bias) in a series of observation obtained in repeated execution of the measurement

NOTE An effect exhibiting a fixed, but random value while executing the measurement repeatedly causes a bias of random value.

3.23
bias
systematic error of the indication of a measuring instrument

[VIM:1993, 5.25]

NOTE A bias of a series of observations about an accepted reference value can be caused either by systematic effects, or by random effects exhibiting (unknown) fixed values in the series of observations.

3.24
representativeness
ability of a series of observations to provide an unbiased estimate of a parameter of a specified statistical population

3.25
population
totality of items under consideration

[ISO 3534-1:2006, 1.1]

NOTE Ensemble of possible results of measurement which can be obtained for a unique measurand by all possible technical realizations of a specified method of measurement.

4 Symbols and abbreviated terms

a	parameter (constant)
b	parameter (constant)
c	parameter (constant)
c_i	sensitivity coefficient
$\mathrm{cov}(x_i, x_k)$	estimate of covariance between input quantities x_i and x_k
$E(X)$	expectation of random variable X
i	index
j	index
k	index
k_p	coverage factor
K	number
L	number of laboratories
M	number
N	number
p	coverage probability; level of confidence
$\sigma(x)$	standard deviation of the population of a random variable X
$s(x)$	experimental standard deviation of data set $x(j)$ with $j = 1$ to N
$t(p,\nu)$	$(1 - p)$-quantile of Student's t-distribution of ν degrees of freedom
u_B	uncertainty caused by bias
$u(x_i)$	standard uncertainty of input value x_i

$u(x_R)$ (combined) standard uncertainty of reference value x_R

$u(y_R)$ (combined) standard uncertainty of reference value y_R

$u(y_R(j))$ (combined) standard uncertainty of reference value $y_R(j)$

$U_p(y)$ expanded uncertainty of result of measurement y on level of coverage p

var(x_i) estimate of the variance of input quantity x_i

var(Y) estimate of the variance of possible results of measurement Y

var(y) estimate of the variance of results of measurement $y(j)$ with j = 1 to N observed in a direct approach

$w(y)$ relative standard uncertainty of a result of measurement y

$W_p(y)$ relative expanded uncertainty of a result of measurement y on level of coverage p

x_i input quantity of the method model equation $y = f(x_1,...,x_K)$

x_R reference value for input quantity x

δX potential deviation of influence quantity x

Y possible result of measurement that could reasonably be attributed to the same measurand by independent replication of the measurement which was executed to obtain the result of measurement y

y result of measurement

y_R accepted value of reference material of the measurand

$y_R(i)$ result of measurement obtained by a reference method of measurement

δY potential deviation of result of measurement y about the (unknown) true value of the measurand, which is not described implicitly by the experimental data to be evaluated

γ level of confidence

μ (unknown) true value of the measurand

ν number of degrees of freedom

ν_{eff} effective number of degrees of freedom

$\chi^2(\gamma, \nu)$ γ–percentile of chi-square distribution of ν degrees of freedom

5 Basic concepts

5.1 Outline

The general objective of this International Standard is to support application of the *Guide to the Expression of Uncertainty in Measurement* (GUM) in the various fields of air quality measurement including ambient air, indoor air, meteorology, stationary source emissions and workplace atmospheres. Standard methods of air quality measurement are considered to be fully documented, e.g. in method standards, standard operating procedures, validation reports or in other technical documents.

Documentation for a given method should comprise

— instructions on intended use (standard operating procedure),

— instructions on correction for systematic effects, if appropriate,

— method model equation $y = f(x_1,..., x_K)$, if results of measurement y are calculated from observed or otherwise known input quantities x_i,

— results of method-validation, if appropriate, and

— instructions on how to assign uncertainty parameters to results of measurement y.

The focus of this International Standard is on how to assign appropriate uncertainty parameters to results of measurement obtained by air quality measurement methods. To this end, uncertainty estimation is considered to be a five-step procedure consisting of

— problem specification (see Clause 6),

— statistical analysis (see Clause 7),

— estimation of variances and covariances (see Clause 8),

— evaluation of uncertainty parameters (see Clause 9), and

— reporting (see Clause 10).

Figure 1 relates this five-step procedure to the eight steps recommended by the GUM.

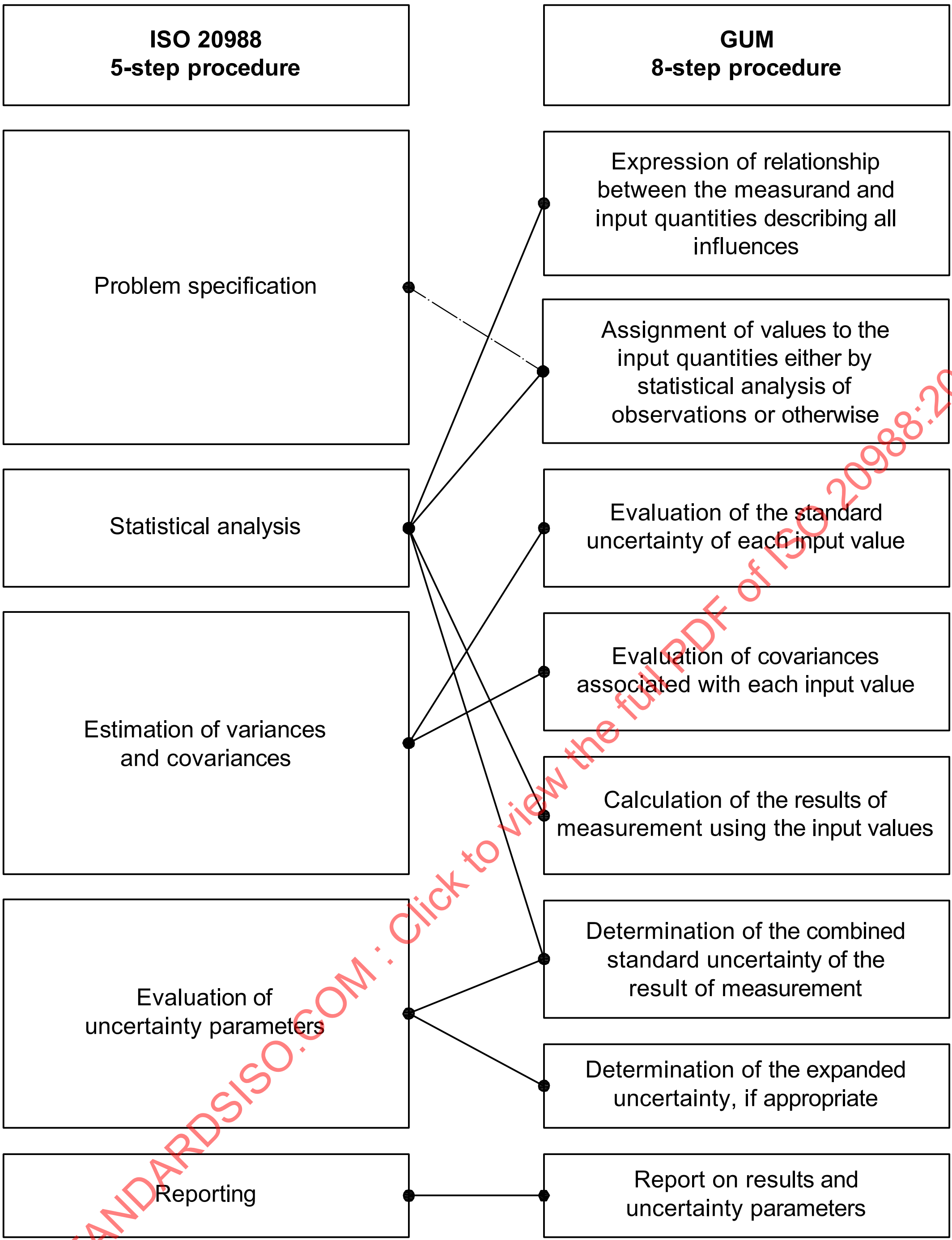


Figure 1 — Comparison of the 5-step ISO 20988 procedure (left side) with the 8-step procedure of the GUM (right side)

The main objectives of problem specification as a separate first step are

— to identify the questions to be answered, and

— to provide input data to be evaluated.

Starting from a proper problem specification, this International Standard provides guidance to statistical analysis and to evaluation methods which are applicable without mathematical expertise. Problem specification requires expert knowledge of technical aspects of the measurement considered and at least a basic understanding of the general statistical concept of uncertainty estimation described by the GUM. A brief introduction to the statistical aspects of uncertainty estimation is provided in 5.2, 5.3 and 5.4.

5.2 Measurement uncertainty

Measurement uncertainty is defined a “parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand” (see 3.1).

An appropriate uncertainty parameter can be:

— the (combined) standard uncertainty $u(y)$ of a result of measurement y;

— the expanded uncertainty $U_p(y)$ of a result of measurement y on a specified level of coverage p.

In accordance with definition 3.1, the (combined) standard uncertainty $u(y)$ of a result of measurement y is the positive square root of an estimate var(Y) of the variance of the population of possible results of measurement Y that could reasonably be attributed to the same measurand by independent replication of the measurement. Accordingly, a basic task in uncertainty estimation is to provide an estimate var(Y) of the variance of the population of possible results of measurement Y. A detailed statistical discussion is provided in Clause 7.

Following definition 3.4, the expanded uncertainty $U_p(y)$ describes an interval $[y - U_p(y); y + U_p(y)]$ about a specific result of measurement y, which is expected to encompass a large fraction p of the possible results that could reasonably be attributed to the same measurand by independent replication of the measurement. For a specified coverage probability p, the corresponding expanded uncertainty $U_p(y)$ is obtained as a multiple of the (combined) standard uncertainty $u(y)$. This implies a Gaussian distribution of possible results of measurement about the unique but unknown value of the measurand. For details, see 9.3.

The common understanding of an uncertainty interval $[y - U_p(y); y + U_p(y)]$ is that of an estimate characterizing the range of values within which the true value of the measurand lies (see ISO/IEC Guide 98:1995, 2.2.4), i.e. within which the value of the measurand is confidently believed to lie [4]. The coverage probability p describes the degree of belief that the true value of the measurand is covered by the interval $[y - U_p(y); y + U_p(y)]$.

Given a specified expanded uncertainty $U_p(y)$ and an appropriate set of input data, the coverage probability p of the uncertainty interval $[y - U_p(y); y + U_p(y)]$ about an observed result of measurement y can be tested in a robust manner. This method does not imply a Gaussian distribution of possible results of measurement about the unknown value of the measurand. Details are given in Annex A.

If appropriate, the combined standard uncertainty $u(y)$ can be described as a function of the result of measurement y, e.g. $w(y) = u(y)/y$ = constant. An uncertainty function of this kind can be closely linked to a method model equation $y = f(x_1,..., x_K)$ used to obtain results of measurement y. This concept is illustrated by Figure 2.

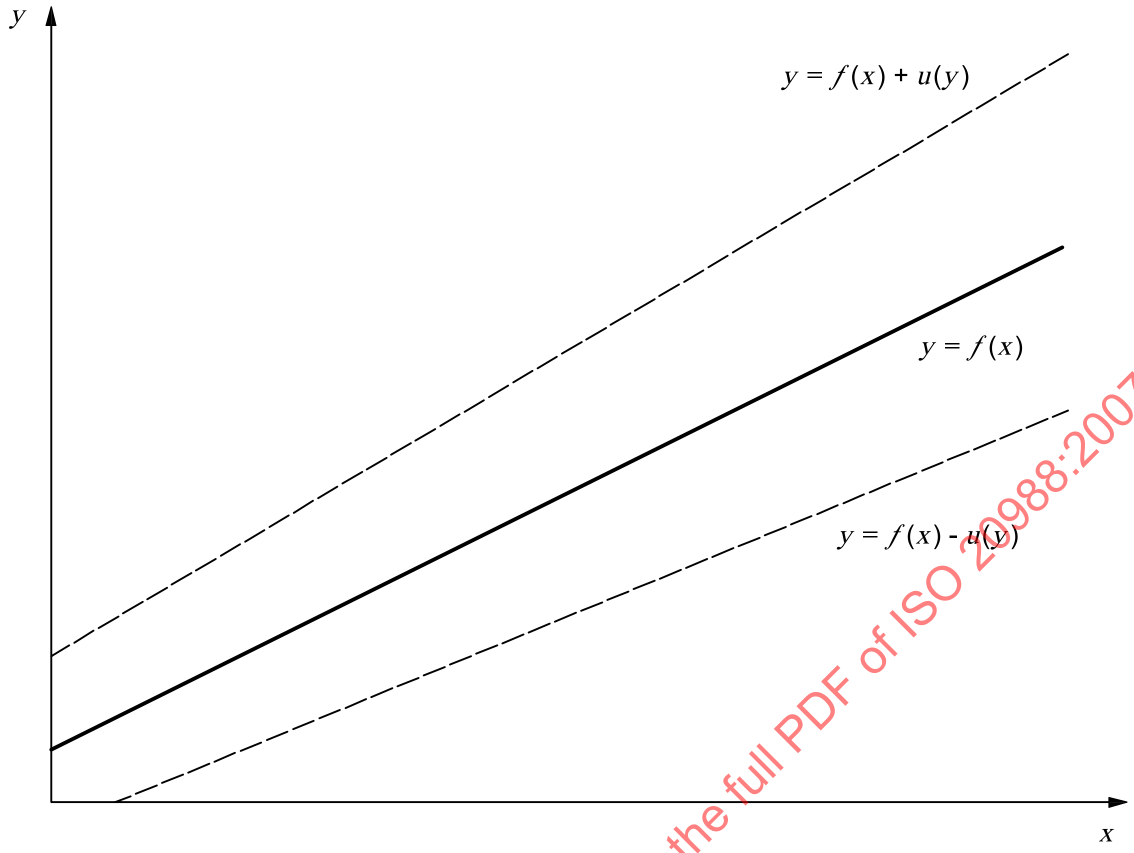


Figure 2 — Method model equation and uncertainty function

It is implicit in this International Standard that an uncertainty parameter obtained by evaluation of a specified set of input data shall be appropriate to predict the uncertainty of future results of measurement obtained by means of the same method of measurement under conditions represented by the input data evaluated. In order to ensure this, it is essential to provide supporting evidence that the evaluated input data are representative of the application of the method of measurement that will produce the results to be qualified by an uncertainty parameter.

5.3 Correction for systematic effects

Correction for systematic effects is an integral part of a measurement as far as required by the method documentation. In general, correction for systematic effects is achieved by comparison with one or more reference standards, e.g. in calibration or in drift control procedures. Appropriate reference standards can be provided by certified reference materials or by certified reference methods of measurement. By comparison with reference materials of SI units, traceability of possible results of measurement can be established. For a reference method being considered a primary measurement standard, comparison with other reference standards is not necessary for the purpose of correction.

It is a general recommendation of the GUM that corrections should be applied for all recognized significant systematic effects (see ISO/IEC Guide 98:1995, 3.2.4). In general, a correction procedure described by the method documentation can exhibit a certain degree of imperfection, e.g. due to its statistical character and due to the uncertainty of the reference standards used for this purpose. As an expression of the imperfection of a correction procedure, a series of corrected results of measurement obtained by the same measuring system can exhibit a residual bias, which is considered a random variable of expected value zero.

If a correction is applied in a measurement by means of a method model equation used to calculate the result of measurement, the uncertainty of the applied correction is taken into account properly.

If a bias is not corrected for, it shall be taken into account as an additional source of uncertainty.

In conclusion, for uncertainty estimation, it is necessary to collect series of observations that allow the user to evaluate both the variations and the bias occurring in the intended use of the method of measurement. If uncorrected significant bias was not taken into account, the estimation of measurement uncertainty is incomplete.

NOTE The terms “effect”, “influence” and “source of uncertainty” are used with synonymous meaning in this International Standard.

5.4 Provision of input data

Input data for uncertainty estimation shall be representative of all effects causing variation or bias in results of measurement. Appropriate input data can be provided either by series of observations, or by external sources, or by expert judgement.

From a practical point of view, uncertainty estimation can be realized either in an indirect approach or in a direct approach.

In an indirect approach, variations and bias are, in a first step, evaluated separately for the input quantities x_i of the method model equation $y = f(x_1,..., x_K)$ used to obtain results of measurement y. For this purpose, estimates of the variances and covariances of the input quantities x_i can be provided by a Type A evaluation of series of observations or by a Type B evaluation based on expert judgement. Finally, a weighted sum of variances and covariances provides the wanted uncertainty estimate.

In a direct approach, the influences of the dominating effects causing variation and bias of the result of measurement y are investigated in a pooled way by comparison with one or more reference values of the measurand. Effects not varied in a direct approach shall be taken into account separately, e.g. by a Type B evaluation based on expert judgement. In a direct approach, the uncertainty estimation can be much simpler than in an indirect approach.

The focus of the GUM is on the indirect approach without excluding the direct approach.

The basic Type A evaluation method described by the GUM requires a series of unbiased observations of the same unchanged measurand obtained by the same measuring system. This experimental design is called simple random sampling. From a practical point of view, simple random sampling requires complete randomization of all effects between repeated observations of the same unchanged measurand. Simple random sampling is rarely realized under conditions of intended use of methods of air quality measurement, mainly due to the potential presence of uncorrected bias. The considerations of the GUM concerning a Type A evaluation are not exhaustive. There are many situations that can be treated by statistical methods different from the basic Type A evaluation described by the GUM (see ISO/IEC Guide 98:1995, 4.2.8).

In air quality measurements, it is often more convenient and cost-effective to provide input data for uncertainty estimation in experimental designs different from simple random sampling. In this International Standard, the following experimental designs are considered:

A1: simple random sampling;

A2: repeated observation of a reference material by a measuring system;

A3: observation of different reference materials in a calibration procedure;

A4: repeated observation of different reference materials by identical measuring systems;

A5: parallel measurements with a reference method of measurement;

A6: paired measurements of two identical measuring systems;

A7: interlaboratory comparison of identical measuring systems;

A8: parallel measurement of identical measuring systems.

The experimental designs of types A1 to A8 are applicable in indirect as well as in direct approaches for uncertainty estimation of methods of air quality measurement, comprising the following:

— methods of measurement corrected for systematic effects by (repeated) observation of reference material;

— methods of measurement evaluated by repeated observation of reference materials of the measurand prior to routine application;

— methods of measurement calibrated by parallel measurement with a reference method of measurement;

— methods of measurement verified by parallel measurement with a reference method of measurement;

— legal or other accepted reference methods of measurement validated by inter-comparison tests.

Appropriate series of observations can be provided, e.g. by one of the following procedures:

— QA/QC procedure applied repeatedly to the measuring system;

— verification procedure applied once to the measuring system;

— evaluation procedure applied to several measuring systems of the same type;

— validation procedure applied once to several measuring systems of the same type;

— another performance test applied to the measuring system.

Input data for uncertainty estimation can also be provided by external sources if these data are based on statistical evaluation of series of observations, such as accepted values and uncertainties of reference materials, values and uncertainties of instrument constants provided by independent reports or values and uncertainties of physical or chemical constants provided by handbooks (see ISO/IEC Guide 98:1995, 4.1.3).

NOTE The use of external data on reproducibility and trueness of a method of measurement that were obtained by application of ISO 5725-2 [5], ISO 5725-3 [6], ISO 5725-4 [7] and ISO 5725-5 [8] is described in ISO/TS 21748 [9].

If input data cannot be provided as a series of observations or from an external source, such data can be obtained by expert judgement and evaluated by a method of Type B.

The applicability of an uncertainty parameter for future results of measurement obtained by the evaluated method of measurement depends on the representativeness of the input data. The degree of representativeness achieved by a set of input data depends on the following:

— effects described by the input data;

— sample size of the collected series of observations;

— uncertainty of the reference standards applied in this investigation.

The closer the input data describe all effects influencing the measurement, and the smaller the uncertainty of the reference standards, the better is the predictive power of an obtained uncertainty parameter for future results of measurement.

Of course, it is an important issue to test the predictive power of an uncertainty parameter, e.g. by another independent evaluation of measurement uncertainty.

For estimating an expanded uncertainty $U_{0,95}(y)$ on a level of confidence of 95 %, it is recommended to provide a series of observations comprising at least 20 applications of the specified method of measurement. Otherwise, the applicability of the obtained uncertainty parameter cannot be subjected to a meaningful test.

For estimating an expanded uncertainty $U_{0,66}(y)$ on a level of confidence of 66 %, it is recommended to provide a series of observations comprising at least seven applications of the specified method of measurement. Otherwise, the applicability of the obtained uncertainty parameter cannot be subjected to a meaningful test.

For details on the provision of input data and the applicable mathematical evaluation methods, see 6.4 and 8.2, respectively.

6 Problem specification

6.1 Objectives

The objective of problem specification in uncertainty estimation is to identify

— the measurement to be considered,

— the required uncertainty parameter,

— the input data to be evaluated, and

— the effects not described by input data.

Figure 3 outlines the relationships between the elements of problem specification in uncertainty estimation. Problem specification requires expert knowledge of technical aspects of the measurement considered. Guidance provided in 6.2 to 6.4 is applicable without expertise in statistical modelling of measuring processes.

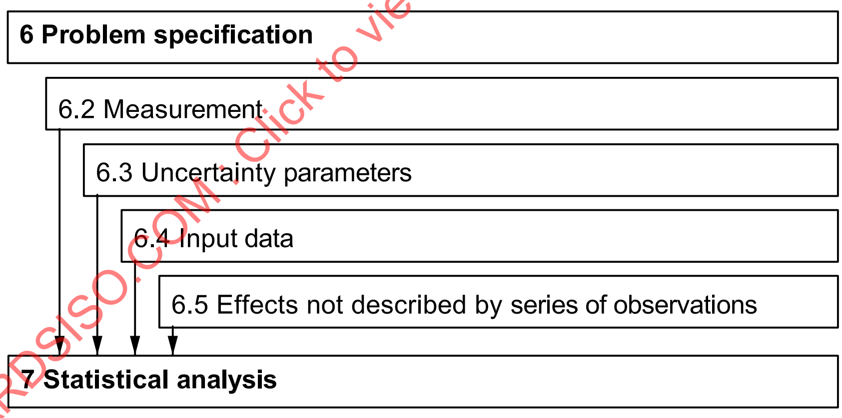


Figure 3 — Elements of problem specification in uncertainty estimation

6.2 Measurement

The measurement shall be specified (at least) in terms of

— measurand,

— method of measurement,

— method model equation $y = f(x_1,..., x_K)$, if results of measurement y are calculated from observed or otherwise known input quantities x_i, and

— intended application of the method of measurement.

The measurand shall be specified in such a way that it is expected to exhibit an unknown, but unique, value μ at least for the time period needed to perform a single measurement.

The measurand is the physical quantity to be assigned a numerical value and a unit of measurement by means of the specified measurement. Furthermore, the measurand shall be specified in such a way that it could be subjected, at least in principle, to more than a single measurement. This is more important for the provision of input data for uncertainty estimation than for routine execution of a considered method of measurement. In air quality, the measurand can change value as a function of time and space.

The method of measurement shall be specified completely, e.g. by

— the applicable procedure, e.g. the standard operating procedure (SOP),

— the type of application (e.g. in routine monitoring of stationary source emissions, in routine monitoring of workplace atmospheres, in routine monitoring of ambient air, or as a reference standard in a laboratory),

— the ambient conditions of that application (e.g. variations in ambient conditions), and

— the conditions of control, (e.g. for calibration or drift control).

Often, appropriate descriptions are available as part of the method documentation.

Additional information on the method of measurement can be provided, e.g. on effects causing variations and bias in the considered application.

If a method model equation $y = f(x_1,..., x_K)$ is used to obtain results of measurement y in intended use from observed or otherwise known input quantities x_i, its mathematical structure shall be known for evaluation of an indirect approach. For an introductory explanation of the direct and the indirect approach, see 5.4.

NOTE 1 In this International Standard, the measurand is understood to be a quantifiable property of an object of measurement. The object of measurement can be, e.g. the gas emitted by a stack of specified cross-section within a specified time period, or the ambient air at a specified sampling location within a defined time interval. The corresponding measurand can be, e.g. the mass (flow) of sulfur dioxide emitted by the specified stack within the specified time period, or the concentration of sulfur dioxide in ambient air at the specified location within the defined time interval.

NOTE 2 The method model equation is sometimes called the “analytical equation” in air quality measurement.

The intended application of the method of measurement shall be specified in such a way that the representativeness of the input data can be assessed properly. This is necessary in order to ensure, that an obtained uncertainty parameter is appropriate to describe results of measurement obtained in the intended application of the method of measurement. In statistical terms, the application of the method of measurement describes the statistical population of possible results of measurement Y to be considered in the uncertainty estimation.

The application of the method of measurement can be specified in different ways, e.g. as follows:

— an application of an individual measuring system under well defined laboratory conditions;

— an application of different measuring systems of the same type operated in a monitoring network by the same laboratory;

— an application of different measuring systems of the same type operated in a wide range of field conditions by different laboratories.

6.3 Uncertainty parameters

The required uncertainty parameter shall be specified. The uncertainty parameter to be provided for the specified population of results of measurement y can be one of the following quantities:

— (combined) standard uncertainty $u(y)$ in units of y;

— relative standard uncertainty $w(y)$;

— expanded uncertainty $U_p(y)$ on a specified level of coverage p in units of y;

— relative expanded uncertainty $W_p(y)$ on a specified level of coverage p.

Uncertainty estimation can be simplified considerably, if a common uncertainty parameter is applicable to a specified range of values y of the result of measurement. For more information on uncertainty parameters, see Clause 9.

6.4 Input data

6.4.1 General

At least the following details on input data for uncertainty estimation shall be described:

— the approach used to investigate variation and bias occurring in intended use of the method of measurement;

— the experimental design(s) used to collect the series of observations;

— the series of observations to be evaluated;

— the representativeness of the series of observations provided as input data.

The approach used to investigate variations and bias can be either an indirect approach or a direct approach.

In a direct approach, input data are obtained in a single experimental design that provides information on deviations and bias by comparison with one or more reference values of the measurand.

In an indirect approach, input data are obtained in different experimental designs for the different input quantities x_i of the method model equation $y = f(x_1,..., x_K)$ used to calculate the result of measurement y.

Table 1 summarizes the experimental designs A1 to A8 considered in this International Standard. Applicable evaluation methods of Type A for these experimental designs are described in Annex B.

Table 1 — Experimental designs and input data

Type	Experimental design	Typical application	Input data	
			Direct approach	Indirect approach
A1	Simple random sampling	Repeated unbiased observation of the same (unknown) measurand by means of the same measuring system	$y(j)$ with j = 1 to N	$x_i(j)$ with j = 1 to N
A2	Repeated observation of a reference material by a measuring system	Drift control procedure applied to the measuring system	$y(j)$ with j = 1 to N y_R	$x_i(j)$ with j = 1 to N $x_{R,i}$
A3	Observation of different reference materials in a calibration procedure	Calibration procedure providing at least 3 repeated observations of each reference materials	$x(j)$ with j = 1 to N $y_R(j)$ with j = 1 to N	$z(j)$ with j = 1 to N $x_R(j)$ with j = 1 to N
A4	Observation of different reference materials by identical measuring systems	Method validation before routine application providing N independent observations of different reference materials	$x(j)$ with j = 1 to N $y_R(j)$ with j = 1 to N	$z(j)$ with j = 1 to N $x_R(j$ with j = 1 to N
A5 Case 1	Parallel measurements with a reference method of measurement	Calibration procedure providing N uncorrected outputs in parallel measurements with a reference method of measurement	$x(j)$ with j = 1 to N $y_R(j)$ with j = 1 to N	$z(j)$ with j = 1 to N $x_R(j)$ with j = 1 to N
A5 Case 2	Parallel measurements with a reference method of measurement	Verification procedure providing N corrected outputs in parallel measurements with a reference method of measurement	$y(j)$ with j = 1 to N $y_R(j)$ with j = 1 to N	$x(j)$ with j = 1 to N $x_R(j)$ with j = 1 to N
A6	Paired measurement of two identical measuring systems	Validation of a reference method of measurement; Validation of a calibrated method of measurement.	$\{y(1, j); y(2, j)\}$ with j = 1 to N	$\{x(1, j); x(2, j)\}$ with j = 1 to N
A7	Interlaboratory comparison of identical measuring systems	Comparison providing N observations of the same measurand by K laboratories using the same method of measurement	$y(k, j)$ with j = 1 to N and k = 1 to K	$x(k, j)$ with j = 1 to N and k = 1 to K
A8	Parallel measurement of identical measuring systems	N parallel measurements with K identical measuring systems	$y(k, j)$ with j = 1 to N and k = 1 to K	$x(k, j)$ with j = 1 to N and k = 1 to K

Experimental designs not mentioned in Table 1 may be evaluated as well for uncertainty estimation, if their execution and statistical evaluation is documented in sufficient detail.

The experimental design(s) used to collect input data shall be documented in sufficient detail to assess the representativeness of the input data for the possible results of measurement to be described by the specified uncertainty parameter. The results of assessing the representativeness of the input data shall be documented.

6.4.2 Assessment of representativeness

For the series of observations provided as input data, it shall be assessed whether in the applied data collecting procedure

a) the method of measurement was operated in accordance with the same standard operating procedure as in the intended application,

b) the ambient conditions covered at least the range of variation expected to occur in the intended application of the method of measurement,

c) the conditions of control of the method of measurement were the same as in the intended application,

d) if appropriate, the reference standards used to correct the method of measurement were of the same quality as those applied in intended use,

e) all parts of the method of measurement were subjected to an experimental design either separately or pooled providing the considered series of observations, and

f) if appropriate, operation of the method of measurement by different laboratories was evaluated in an experimental design.

If the statements a) to f) are fulfilled appropriately, the provided series of observations are considered representative for the specified uncertainty estimation. Statement d) applies to methods of measurement requiring correction for systematic effects. Statement f) applies, if the uncertainty parameter shall describe results of measurement (to be) obtained by different laboratories in application of the specified method of measurement.

If one or more of the statements a) to f) are not all fulfilled appropriately, additional input is needed to provide representative input data. Additional input data can be provided either by provision of more appropriate series of observations, or by expert judgement as described in 6.5.

6.5 Effects not described by series of observations

If an effect causing variations or bias is not described in a representative way by a series of observations, although it is expected to influence possible results of measurement, this effect shall be described separately, e.g. by an additional deviation δY_j of the result of measurement. The numerical values of such an additional deviation shall be assessed by expert judgement. The basic task of this expert judgement is the provision of an estimate of the maximum range [$\min(\delta Y_j) \leqslant \delta Y_j \leqslant \max(\delta Y_j)$] of the deviation δY_j. This information is needed to provide an estimate $\mathrm{var}(\delta Y_j)$ of the variance of the deviation δY_j. Details on variance estimation based on expert judgement (Type B evaluation) are provided in 8.3. A Type B evaluation should only supplement a Type A evaluation.

Instead of estimating the maximum value, $\max(\delta Y_j)$, of a potential deviation δY_j directly, it can sometimes be more convenient to estimate it indirectly by means of Equation (1):

$$\max(\delta Y_j) = c_j \cdot \max(\delta X_j) \qquad (1)$$

In the latter case, δX_j designates a potential deviation of the considered influence quantity x_j from a specified reference value, and c_j the sensitivity coefficient of the method of measurement with respect to changes of that influence quantity.

NOTE The sensitivity coefficient c_j can be determined from separate investigation, e.g. within a method validation study.

Typical effects which can sometimes not be described in a representative way by a series of observations are summarized in Table 2 (this list of effects is nonexhaustive).

An additional deviation δY_j may be neglected, if the corresponding variance estimate $\mathrm{var}(\delta Y_j)$ contributes less than 5 % to the variance estimate $\mathrm{var}(Y)$ used in the uncertainty estimation.

Table 2 — Effects which can require separate assessment

No.	Effect	Assessment of possible influence on the result of measurement
1	Sampling efficiency F_{sam} not corrected for by calibration procedure	$\delta \Psi_{sam} = y\,(1 - F_{sam})$
2	Extraction efficiency F_{ext}	$\delta \Psi_{ext} = y\,(1 - F_{ext})$
3	Variation of ambient temperature T	$\delta Y_T = c_T\, \delta T$
4	Variation of ambient pressure P	$\delta Y_P = c_P\, \delta P$
5	Variation of ambient humidity H	$\delta Y_H = c_H\, \delta H$
6	Variation of composition of air sampled Y_{INT}	$\delta Y_{INT} = c_{INT}\, Y_{INT}$
7	Variation of line voltage V	$\delta Y_V = c_V\, \delta V$

7 Statistical analysis

7.1 Objectives

The objective of statistical analysis in uncertainty estimation is to provide a mathematical rational for calculating an estimate var(Y) of the variance of the population of possible results of measurement Y that could reasonably be attributed to the same measurand by independent replication of the measurement which was executed to obtain the result of measurement y. Based on that variance estimate, the (combined) standard uncertainty $u(y)$ of the result of measurement y is finally calculated by $u(y) = \sqrt{\text{var}(Y)}$.

This International Standard provides guidance to statistical analysis applicable without expertise in statistical modelling of measuring processes. Statistical modelling in the framework of uncertainty estimation is simplified considerably by separate consideration of the indirect and the direct approach. Whereas the recommendations of the GUM are addressed explicitly to the more complex indirect approach, they apply as well to the mathematically much simpler direct approach. In contrast to the indirect approach, a direct approach starts from a single series of observed results of measurement provided as input data.

The appropriate statistical model equation depends on

— the approach used to investigate variations and bias occurring in the method of measurement,

— the experimental design(s) used in that approach,

— the series of observations provided as input data, and

— the representativeness of the input data.

Based on the statistical model equation, an appropriate equation for estimating var(Y) is obtained by the rule of uncertainty propagation.

If the uncertainty parameter is the coverage probability p of a specified interval $[y - U_p(y); y + U_p(y)]$ about a result of measurement y, statistical analysis can be simplified considerably as described in 9.3.

Table 3 emphasizes the differences in the 5-step procedure of uncertainty estimation in the direct and indirect approach.

Table 3 — Elements of uncertainty estimation in direct and indirect approach

Step	Element	Direct approach	Indirect approach
1	**Problem specification**		
	Experimental design	one experimental design, e.g. of type A1 to A8	more than one experimental design, e.g. of Type A1 to A8
	Input data	one series of observations and reference values of the measurand y See 6.4.	series of observations of input quantities x_i or estimates x_i and var(x_i) for each input quantity x_i See 6.4.
	Additional deviation	$\min(\delta Y_j) \leqslant \delta Y_j \leqslant \max(\delta Y_j)$, if appropriate	$\min(\delta Y_j) \leqslant \delta Y_j \leqslant \max(\delta Y_j)$, if appropriate
2	**Statistical analysis**		
	Method model equation	$y = f(x_1, x_2, \ldots)$, if available	$y = f(x_1, x_2, \ldots)$
	Statistical model equation	$Y = y + \delta Y$	$Y = f(x_1, x_2, \ldots) + \delta Y$
	Variance equation	$\mathrm{var}(Y) = \mathrm{var}(y) + \mathrm{var}(\delta Y)$ See 7.3.	$\mathrm{var}(Y) = c_1^2\,\mathrm{var}(x_1) + c_2^2\,\mathrm{var}(x_2) + \ldots$ $+2\,c_1 c_2\,\mathrm{cov}(x_1, x_2) + \ldots$ $+\mathrm{var}(\delta Y)$ See 7.2.
3	**Estimation of variances and covariances**		
	Type A	$\mathrm{var}(y) = u^2(y)$ See 8.2 and Annex B.	$\mathrm{var}(x_i) = u^2(x_i)$ See 8.2 and Annex B.
	Type B	$\mathrm{var}(\delta Y) = \ldots..$ See 8.3.	$\mathrm{var}(\delta Y) = \ldots..$ See 8.3.
	Covariances	See 8.4.	See 8.4.
4	**Evaluation of uncertainty parameters**		
	(Combined) standard uncertainty	$u(y) = \sqrt{\mathrm{var}(Y)}$	$u(y) = \sqrt{\mathrm{var}(Y)}$
	Expanded uncertainty	$U_p(y) = k_p(\nu) \cdot u(y)$ See 9.2.	$U_p(y) = k_p(\nu) \cdot u(y)$ See 9.2.
5	**Reporting**		

Although Y is called the measurand by the GUM, the meaning of Y in the mathematical context of the GUM is that of a random variable describing a “possible result of measurement”. The character Y shall not be confused with the unknown but unique value μ of the measurand under observation. The general concept of measurement implies that the measurand exhibits a unique value at least for the time interval described by an individual result of measurement.

7.2 Indirect approach

In an indirect approach, variation and bias are evaluated, in a first step, separately for the input quantities x_i of the method model equation $y = f(x_1, \ldots, x_K)$ used to obtain results of measurement y. The following data can be used as input data:

— series of observations of input quantities x_i collected, e.g. in experimental designs of types A1 to A8;

— estimates of x_i and var(x_i) provided by external sources, such as quantities associated with calibrated measurement standards, certified reference materials, reference data obtained from handbooks and data on reproducibility and trueness.

NOTE The use of external data on reproducibility and trueness for uncertainty estimation is described in ISO/TS 21748 [9].

Additional deviations δY_j can be caused by effects not represented by a series of observations. If necessary, these deviations can be assessed by expert judgement and a Type B evaluation.

Under these conditions, an appropriate statistical model equation for uncertainty estimation is provided in generic terms by Equation (2):

$$Y = f(x_1,\ldots,x_K) + \sum_{j=1}^{M} \delta Y_j \quad (2)$$

where

Y is a possible result of measurement;

x_i is the input quantity of the method model equation $y = f(x_1,\ldots, x_K)$ used to calculate the result of measurement y;

δY_j is an additional deviation of result of measurement y not represented by the series of observations of input quantities x_i;

K is the number of input quantities of the method model equation;

M is the number of additional deviations to be assessed by a Type B evaluation.

An additional deviation δY_j shall be taken into account in the statistical model equation given by Equation (2), if the corresponding variance var(δY_j) makes up at least 5 % of the population variance var(Y) described in Equation (3). Otherwise, the deviation δY_j may be neglected in the uncertainty estimation.

In a second step, the rule of uncertainty propagation shall be applied to the statistical model equation given by Equation (2). In this way, the variance estimate var(Y) is obtained from variance (budget) equation as given by Equation (3):

$$\mathrm{var}(Y) = \sum_{i=1}^{K} c_i^2\, \mathrm{var}(x_i) + 2\sum_{i=1}^{K} \sum_{j=i+1}^{K} c_i\, c_j\, \mathrm{cov}(x_i, x_j) + \sum_{j=1}^{M} \mathrm{var}(\delta Y_j) \quad (3)$$

where

var(Y) is the estimate of the variance of possible results of measurement Y;

c_i is the sensitivity coefficient with respect to variations of input quantity x_i;

var(x_i) is an estimate of the variance of input quantity x_i;

cov(x_i, x_j) is an estimate of covariance between input quantities x_i and x_j;

var(δY_j) is an estimate of the variance of additional deviation δY_j.

In formal terms, the sensitivity coefficient c_i is the partial derivative of the method model function $y = f(x_1,\ldots, x_K)$ by Equation (4):

$$c_i = \frac{\partial f(x_1,\ldots,x_K)}{\partial x_i} \quad (4)$$

In practical terms, a sensitivity coefficient c_i can be obtained in different ways:

— by application of rules of algebra;

— by a numerical calculation of the derivative;

— as the mean value of the ratio of observed changes $\Delta y(j)$ of the result of measurement divided by the change $\Delta x_i(j)$ causing the observed change $\Delta y(j)$ as given by Equation (5):

$$c_i = \sum_{j=1}^{N} \frac{[\Delta y(j)/\Delta x_i(j)]}{N} \tag{5}$$

The variance estimates var(x_i) in Equation (3) shall be evaluated either by a Type A evaluation method appropriate for the experimental design used to obtain the input data or by reference to external sources. Type A methods for the evaluation of the experimental designs A1 to A8 are described in detail in Annex B.

Variance estimates var(δY_j) of deviations δY_j shall be calculated by a Type B evaluation method described in 8.3.

Covariances cov(x_i, x_j) shall be considered zero if the series of observations provided for the input quantities x_i and x_j were obtained independent of each other in different experimental designs. Otherwise, estimates of covariances cov(x_i, x_j) shall be calculated by a method described in 8.4.

7.3 Direct approach

In a direct approach, variation and bias of results of measurement y are assessed directly in a pooled way in a single experimental design, e.g. of types A1 to A8. Input data are a single series of observations and the corresponding reference values. If necessary, additional deviations δY_j caused by effects not represented by the series of results of measurement are assessed by expert judgement.

Under these conditions, the statistical model equation for uncertainty estimation is given by Equation (6):

$$Y = y + \sum_{j=1}^{M} \delta Y_j \tag{6}$$

where

Y is a possible result of measurement;

y is a result of measurement provided as input data (see 6.4);

δY_j is an additional deviation of result of measurement y not represented by the series of results of measurement;

M is the number of additional deviations to be assessed by a Type B evaluation.

An additional deviation δY_j shall be taken into account in the statistical model equation given by Equation (6), if the corresponding variance var(δY_j) makes up at least 5 % of the estimate var(Y) of the population variance described in Equation (7). Otherwise, the deviation δY_j may be neglected in uncertainty estimation.

The rule of uncertainty propagation shall be applied to the statistical model equation given by Equation (6). In this way, the variance estimate var(Y) is obtained from the variance (budget) equation as given by Equation (7):

$$\mathrm{var}(Y) = \mathrm{var}(y) + \sum_{j=1}^{M} \mathrm{var}(\delta Y_j) \tag{7}$$

where

var(Y) is the estimate of the variance of possible results of measurement Y;

var(y) is an estimate of the variance of the series of results of measurement y;

var(δY_j) is an estimate of the variance of additional deviation δY_j obtained by a Type B evaluation.

The variance estimate var(y) in Equation (7) shall be calculated by a Type A evaluation method appropriate for the experimental design used to obtain the input data. Type A methods for the evaluation of the experimental designs A1 to A8 are described in detail in Annex B.

Variance estimates var(δY_j) of deviations δY_j not described by the series of results of measurement shall be obtained by a Type B evaluation described in 8.3.

NOTE Each direct approach can be considered a special case of an indirect approach.

7.4 Statistical validity

The statistical validity of the variance estimate var(Y) or the corresponding standard uncertainty $u(y) = \sqrt{\mathrm{var}(Y)}$ obtained in accordance with 7.2 or 7.3 is described by an effective number of degrees of freedom ν_{eff}.

In general, the effective number of degrees of freedom ν_{eff} of the variance estimate var(Y) can be obtained by Welch-Satterthwaite Equation (8a) for an indirect approach and by Equation (8b) for a direct approach:

$$\nu_{\mathrm{eff}} = \frac{\mathrm{var}^2(Y)}{\sum_{i=1}^{K} \frac{c_i^4 \, \mathrm{var}^2(x_i)}{\nu_i} + \sum_{j=1}^{M} \frac{\mathrm{var}^2(\delta Y_j)}{\nu_j}} \tag{8a}$$

$$\nu_{\mathrm{eff}} = \frac{\mathrm{var}^2(Y)}{\frac{\mathrm{var}^2(y)}{\nu_y} + \sum_{j=1}^{M} \frac{\mathrm{var}^2(\delta Y_j)}{\nu_j}} \tag{8b}$$

where

ν_{eff} is the effective number of degrees of freedom assigned to variance estimate var(Y);

ν_i is the (effective) number of degrees of freedom assigned to variance estimate var(x_i);

ν_j is the (effective) number of degrees of freedom assigned to variance estimate var(δY_j).

If additional deviations δY_j not described by the series of observations provide insignificant contributions to the variance estimate var(Y), they may be neglected in Equation (8a) and Equation (8b), respectively.

Non-integer values obtained for ν_{eff} shall be rounded downward to the next integer value.

Sometimes, the general structure of a variance estimate obtained by evaluation of a data set $x(j)$ with j = 1 to N can be given by Equation (9)

$$\mathrm{var}(x) = s^2(x) + u_\mathrm{B}^2 \tag{9}$$

where $s(x)$ is the experimental standard deviation and u_B is a bias term. In this case, the effective number of degrees of freedom ν assigned to the variance estimate var(x) can be obtained in good approximation as follows:

— by the number of independent observations used to calculate $s(x)$, if the contribution of the standard deviation $s(x)$ to the variance estimate var(x) is at least 50 %, i.e. if $s^2(x)/\mathrm{var}(x) \geqslant 0{,}5$;

— by the number of independent data used to estimate the bias u_B, if the contribution of the bias u_B to the variance estimate var(x) is at least 50 %, i.e. if $u_\mathrm{B}^2/\mathrm{var}(x) \geqslant 0{,}5$.

8 Estimation of variances and covariances

8.1 General

In order to calculate a variance estimate var(Y) by means of the variance equation obtained for a direct or for an indirect approach by application of Clause 7, estimates of the variances and covariances contributing to the variance equation shall be obtained as follows:

— by statistical evaluation of series of observations (a so-called Type A evaluation);

— by expert judgement (a so-called Type B evaluation).

8.2 Variance estimates of Type A

The applicable evaluation method of Type A depends on the experimental design used to collect the series of observations to be evaluated. Although focussed on consideration of simple random sampling (experimental design A1), the general recommendations of the GUM apply as well to experimental designs such as A2 to A8.

Annex B provides Type A evaluation methods applicable to estimate variances from series of observations obtained in experimental designs of type A1 to A8. The application of these evaluation methods is recommended.

This International Standard does not exclude application of other evaluation methods for estimating variances. Other statistical methods may be applied, if they are well documented and consistent with the GUM.

NOTE 1 The use of repeatability, reproducibility and trueness estimates in measurement uncertainty estimation is described in ISO/TS 21748.

NOTE 2 A reproducibility standard deviation s_R obtained by evaluation of an interlaboratory comparison study according to ISO 5725-2 or by evaluation of a split level design in accordance with ISO 5725-5 can be used to provide a variance estimate var(y) = s^2_R within a direct approach, if the data set evaluated describes the dominant effects influencing the considered method of measurement.

8.3 Variance estimates of Type B

An additional deviation δY_j of the result of measurement y which is not included in the experimental data can be treated in a conservative manner based on available information on

— the expected range of variation [min(δY_j); max(δY_j)] of the deviation δY_j, and

— the expected type of the statistical population of δY_j.

Based on input data of this kind, a conservative estimate of the variance of the deviation δY_j is obtained by Equation (10):

$$\mathrm{var}(\delta Y_j) = \frac{\left(\max(\delta Y_j) + \min(\delta Y_j)\right)^2}{4} + \frac{\left(\max(\delta Y_j) - \min(\delta Y_j)\right)^2}{12} \qquad (10)$$

In the case of $\min(\delta Y_j) = -\max(\delta Y_j)$, Equation (10) simplifies to Equation (11):

$$\mathrm{var}(\delta Y_j) = \frac{\left(\max(\delta Y_j)\right)^2}{3} \qquad (11)$$

If appropriate, a deviation δY_i of the result of measurement y can be related to a deviation δX_i of an observable influence quantity x_i by means of a known sensitivity coefficient c_i as described by $\delta Y_i = c_i\, \delta X_i$. In this way, estimating the range of variation of δY_i can be replaced by estimating the range of variation of δX_i. Examples of Type B evaluation are provided in Table 4.

Table 4 — Examples of Type B evaluation

Statistical population	**Range** $\max(\delta Y_j) = -\min(\delta Y_j)$	**Estimated variance** $\mathrm{var}(\delta Y_j)$
Rectangular	a	$a^2/3$
Triangular	a	$a^2/6$

Evaluation of measurement uncertainty should be based on observed experimental data to the maximum extent possible, as emphasized in ISO/IEC Guide 98:1995, 3.4.1 and ISO/IEC Guide 98:1995, 3.4.2.

8.4 Estimation of covariances

The covariance associated with the values x_i and x_k assigned to two input quantities of the method model equation shall be zero if one of the following conditions is met:

a) x_i and x_k have not been obtained in the same experimental design;

b) either x_i or x_k was kept constant, when the other quantity was repeatedly observed.

NOTE 1 For comparison, see ISO/IEC Guide 98:1995, F.1.2.1.

Accordingly, calculation of covariances can often be avoided by making the appropriate choice of experimental designs providing observed data for uncertainty estimation.

If two deviations δY_i and δY_k are expected to be correlated positively, they may be replaced by a single deviation δY_{ik} with a maximum given by $\max(\delta Y_{ik}) = \max(\delta Y_i) + \max(\delta Y_k)$. In this way, a positive covariance between two deviations δY_i and δY_k is taken into account in a conservative manner.

If two deviations δY_i and δY_k are expected to be correlated negatively, they shall be treated as if they were not correlated. In this way, a negative covariance between two deviations δY_i and δY_k is taken into account in a conservative manner.

If experimental data $\{x_i(j), x_k(j)\}$ with j = 1 to N of two input quantities x_i and x_k of the method model equation $y = f(x_1,..., x_K)$ were observed simultaneously in an experimental design providing comparison with appropriate reference values x_{Ri} and x_{Rk}, an unbiased estimate of the covariance cov(x_i, x_k) shall be obtained by Equation (12):

$$\mathrm{cov}(x_i, x_k) = \sum_{j=1}^{N} \frac{\left(x_i(j) - x_{\mathrm{R}i}\right)\left(x_k(j) - x_{\mathrm{R}k}\right)}{N} + \mathrm{cov}(x_{\mathrm{R}i}, x_{\mathrm{R}k}) \tag{12}$$

Covariance cov(x_{Ri}, x_{Rk}) is an estimate of the correlation between the reference values x_{Ri} and x_{Rk} which is zero, if the reference values x_{Ri} and x_{Rk} have been determined independently of each other.

In the case that each of the series of observations $x_i(j)$ and $x_k(j)$ with j = 1 to N itself is unbiased, the reference values x_{Ri} and x_{Rk} shall be replaced by the sample mean values $\bar{x}_i$ and $\bar{x}_k$ giving the estimate found by Equation (13):

$$\mathrm{cov}(x_i, x_k) = \sum_{j=1}^{N} \frac{\left(x_i(j) - \bar{x}_i\right)\left(x_k(j) - \bar{x}_k\right)}{N-1} \tag{13}$$

where

$$\bar{x}_i = \sum_{j=1}^{N} \frac{x_i(j)}{N}$$

$$\bar{x}_k = \sum_{j=1}^{N} \frac{x_k(j)}{N}$$

The covariance between the two mean values $\bar{x}_i$ and $\bar{x}_k$ obtained from independent series of observations shall be estimated by Equation (14):

$$\mathrm{cov}(\bar{x}_i, \bar{x}_k) = \sum_{j=1}^{N} \frac{\left(x_i(j) - \bar{x}_i\right)\left(x_k(j) - \bar{x}_k\right)}{N(N-1)} \tag{14}$$

NOTE 2 If a data set $x(j)$ with j = 1 to N is obtained by repeated observation of the same reference material of fixed value x_R, the covariance between x_R and the observed deviations d$x(j)$ = $x(j)$ − x_R is estimated properly by $\mathrm{cov}(x_\mathrm{R}, \mathrm{d}x(j)) = 0$.

9 Evaluation of uncertainty parameters

9.1 Objective

The measurement uncertainty of a result y obtained by a specified method of measurement can be quantified either by a combined standard uncertainty $u(y)$ or by an expanded uncertainty $U_p(y)$ on a specified level of coverage p.

9.2 Combined standard uncertainty

The (combined) standard uncertainty $u(y)$ of a result of measurement y obtained by the specified method of measurement shall be calculated by Equation (15):

$$u(y) = \sqrt{\mathrm{var}(Y)} \tag{15}$$

where var(Y) is a variance estimate that is calculated for the result of measurement y by means of the applicable variance equation (see Clause 7).

The relative standard uncertainty $w(y)$ of a result of measurement y shall be calculated by means of Equation (16):

$$w(y) = u(y)/y \tag{16}$$

If the standard uncertainty $u(y)$ is obtained exclusively by a Type A evaluation, a confidence limit for the unknown (true) value estimated by $u(y)$ can be obtained by Equation (17) for a level of confidence γ:

$$L_\gamma(u(y)) = \sqrt{\nu_{\text{eff}} / \chi^2(\gamma, \nu_{\text{eff}})} \cdot u(y) \tag{17}$$

where $\chi^2(\gamma, \nu_{\text{eff}})$ is the γ-percentile of the chi-square distribution of ν_{eff} degrees of freedom.

An upper confidence limit for the true value $\sigma(y)$ of the standard uncertainty is obtained by multiplying $u(y)$ with the appropriate factor, e.g. with 1,27 for γ = 0,90 and ν_{eff} = 20. In this case, the risk of the true value $\sigma(y)$ of the standard uncertainty to exceed 1,27 · $u(y)$ is 10 %.

Table 5 — γ-percentile of the chi-square distribution of ν_{eff} degrees of freedom

ν_{eff}	$\sqrt{\nu_{\text{eff}} / \chi^2(\gamma, \nu_{\text{eff}})}$			
	γ = 0,05	γ = 0,50	γ = 0,90	γ = 0,95
5	0,67	1,07	1,76	2,09
10	0,74	1,03	1,43	1,59
15	0,77	1,02	1,32	1,44
20	0,80	1,02	1,27	1,36
30	0,83	1,01	1,21	1,27
40	0,85	1,01	1,17	1,23
50	0,86	1,01	1,15	1,20
60	0,87	1,01	1,14	1,18
70	0,88	1,00	1,12	1,16
80	0,89	1,00	1,12	1,15
100	0,90	1,00	1,10	1,13
150	0,91	1,00	1,08	1,11

9.3 Expanded uncertainty

9.3.1 General

The expanded uncertainty $U_p(y)$ of the result of measurement y with coverage probability p shall be calculated by multiplication of the (combined) standard uncertainty $u(y)$ and a coverage factor k corresponding to the coverage probability p as described by Equation (18):

$$U_p(y) = k \cdot u(y) \tag{18}$$

The expanded uncertainty $U_p(y)$ describes an interval $[y - U_p(y); y + U_p(y)]$ about the result of measurement y that is expected to encompass a large fraction p of the distribution of values that could reasonably be attributed to the measurand. The fraction p is called coverage probability or level of confidence of the interval $[y - U_p(y); y + U_p(y)]$.

The relative expanded uncertainty $W_p(y)$ of the result of measurement y with coverage probability p is obtained by Equation (19):

$$W_p(y) = k \cdot w(y) \tag{19}$$

The coverage factor k and the coverage probability p shall be stated when reporting an expanded uncertainty $U_p(y)$ of a result of measurement y. Typical coverage factors are $k = 2$ or $k = 3$.

Concerning the relationship between coverage factor k and coverage probability p, the following cases shall be distinguished:

a) The result of measurement y is obtained as mean value of $N > 1$ independent observations y_i of the same measurand of fixed value by means of the same measuring system. The distribution of possible results of measurement Y about the unknown value of the measurand is Gaussian to a good approximation. The standard uncertainty $u(y)$ is estimated by evaluation of both the input data y_i used to obtain the result of measurement y and of additional input data obtained, e.g. from separate evaluations or from external sources. The effective number of degrees of freedom of the uncertainty estimate $u(y)$ is ν.

b) The result of measurement y is obtained by single application of the specified method of measurement. The distribution of possible results Y about the unknown value of the measurand is Gaussian to a good approximation. The standard uncertainty $u(y)$ is estimated only by evaluation of input data obtained separate from the measurement delivering the result y to be qualified by an uncertainty interval. The effective number of degrees of freedom of the uncertainty estimate $u(y)$ is ν.

c) The result of measurement y is obtained by single application of the specified method of measurement. The distribution of possible results Y about the unknown value of the measurand is not described properly by a Gaussian distribution.

Cases a) and b) are considered in 9.3.2. Case c) can be addressed tentatively as described in 9.3.2 if the resulting expanded uncertainty is subjected to a robust test of the assigned coverage probability as described in Annex A.

9.3.2 Expanded uncertainty of results exhibiting a Gaussian distribution

If the distribution of possible results of measurement Y can be described by a Gaussian distribution to a good approximation and an estimate $u(y)$ of the standard deviation of this Gaussian distribution is available with ν degrees of freedom, the relationship between coverage factor k and coverage probability p of uncertainty interval $[y - k \cdot u(y); y + k \cdot u(y)]$ shall be determined by Equation (20):

$$k = t(p, \nu) \tag{20}$$

where

$t(p, \nu)$ is the $(1 - p)$-quantile of Student's t-distribution of ν degrees of freedom;

p is the coverage probability of interval $[-t(p, \nu); +t(p, \nu)]$ by Student's t-distribution with ν degrees of freedom;

ν is the number of degrees of freedom, $\nu = N - 1$ assigned to the standard uncertainty $u(y)$ of a result of measurement y.

In this case, the uncertainty interval $[y - k \cdot u(y); y + k \cdot u(y)]$ can be interpreted as an interval encompassing the unknown value μ of the measurand on a level of confidence described (approximately) by p. This interpretation applies best, if the (combined) standard uncertainty $u(y)$ is obtained exclusively by Type A evaluations.

Table 6 provides coverage factors k for typical coverage probabilities p obtained by Student's t-distribution.

Table 6 — Coverage factor $k = t(p, \nu)$ as a function of coverage probability p and number of degrees of freedom ν obtained from Student's t-distribution

ν	k		
	p = 90 %	p = 95 %	p = 99 %
5	2,02	2,57	4,03
6	1,94	2,45	3,71
7	1,89	2,36	3,50
8	1,86	2,31	3,36
9	1,83	2,26	3,25
10	1,81	2,23	3,17
12	1,78	2,18	3,05
14	1,76	2,14	2,98
16	1,75	2,12	2,92
18	1,73	2,10	2,88
20	1,72	2,09	2,85
30	1,70	2,04	2,75
∞	1,645	1,96	2,58

If a single estimate of an expanded uncertainty $U_p(y)$ is repeatedly attributed to future results $y' \cong y$ by means of relationship $U_p(y') = U_p(y = y')$, the common coverage probability π of all intervals $[y' - U_p(y'); y' + U_p(y')]$ can differ slightly from the required coverage probability p. The risk α of the true coverage probability π of intervals $[y' - U_p(y'); y' + U_p(y')]$ being smaller than a required value p can be assessed by means of Annex A.

NOTE 1 For $\nu \geqslant 30$, an uncertainty interval $[y - 2{,}0 \cdot u(y); y + 2{,}0 \cdot u(y)]$ is assigned a level of confidence of 95 % (or more) to encompass the wanted value of the measurand.

NOTE 2 For $\nu \geqslant 30$, the probability that a (future) result of measurement y exhibits a deviation of more than $2{,}0 \cdot u(y)$ from the wanted true value of the measurand is not exceeding 5 %.

NOTE 3 If an estimate $u(y)$ is replaced by an upper γ confidence limit $L_\gamma(u(y))$, a γ confidence limit on expanded 95 % uncertainty is obtained by $L_\gamma(U_{0,95}(y)) = 1{,}96 \cdot L_\gamma(u(y))$.

10 Reporting

A report on execution of a specified task of uncertainty estimation shall include (at least) the following items:

a) problem specification including the following:

1) method of measurement;

2) required uncertainty parameter;

3) statistical population of possible (future) results of measurement;

4) input data and experimental design(s) applied;

5) representativeness of the input data;

6) effects not described by input data;

b) statistical analysis describing the applied statistical model equation and the variance (budget) equation;

c) evaluation methods describing the applied methods for estimating variances and covariances of input data;

d) obtained numerical value(s) assigned to the uncertainty parameter(s) as well as its range of application.

A clear specification of the range of application of a reported uncertainty parameter is important to avoid misleading usage.

Annex A
(informative)

Testing a coverage probability

A.1 General

Following the definition of expanded uncertainty $U_p = k \cdot u$, coverage probability p can be seen as the fraction of the distribution of values attributable to the measurand which is encompassed by the interval $[y - U_p; y + U_p]$ about the considered result of measurement y.

In a context of elementary statistics, coverage probability p is equivalent to the fraction of the distribution of possible results Y obtainable instead of the observed result of measurement y which is encompassed by the interval $[\mu - U_p; \mu + U_p]$ about the unique but unknown value μ of the measurand. Here, the value μ is considered to coincide with the population mean of the random variable Y. The latter concept is used by the GUM to relate a coverage probability p = 0,95 to the expanded uncertainty $U_p = 2 \cdot u$ in the case of a Gaussian distribution of possible results.

In practical terms, coverage probability p can be related closely to the fraction of observations $y(j)$ of a reference material encompassed by the interval $[y_R - U_p; y_R + U_p]$ about the value y_R of the observed reference material. By statistical inference, an estimate of coverage probability p can be used as well to predict the fraction of future observations of the same reference material which are encompassed by the interval $[y_R - U_p; y_R + U_p]$ about the value y_R. Of course, this prediction is subject to statistical error.

In conclusion, the meaning of an expanded uncertainty $U_p = k \cdot u$ and the corresponding coverage probability p are not restricted to a specific result of measurement y, but can be used as well to predict uncertainty intervals $[y' - U_p; y' + U_p]$ of coverage probability p about future observations y' obtained by means of the same method of measurement. The uncertainty of this predictive use can be described by the standard error $s(p)$ of the estimated value of coverage probability p.

Robust statistics allow the estimation of both the coverage probability p and the corresponding standard error $s(p)$. In addition, robust statistics work without the need to assume a Gaussian error distribution. Finally, hypothesis testing is realized easily within this framework. The only prerequisite for all this is a series of input data provided by a direct approach, e.g. by one of the following procedures:

— observation of a reference material of the measurand (Type A2);

— observation of different reference materials in a calibration procedure (Type A3);

— observation of different reference materials in an evaluation procedure (Type A4);

— parallel measurements with a reference method (Type A5).

A.2 Robust estimation of coverage probability

In the following, the robust estimation of coverage probability is illustrated for a series of N repeated observations $y(j)$ of a single reference material of certified value y_R obtained by the same method of measurement. The value of the expanded uncertainty $U_p = k \cdot u$ or $U_p = \Delta \cdot y$ is provided, e.g. by evaluation of the same series of observations $y(j)$ or by provision of a data quality objective Δ.

With M of the N repeated observations $y(j)$ fulfilling relationship $y_R - U_p \leqslant y(j) \leqslant y_R + U_p$, a robust estimate of the corresponding coverage probability is given by $p = M/(N + 1)$. Accordingly, if all of N observations $y(j)$ fulfil the relationship, a robust estimate of coverage probability is given by $p = N/(N + 1) < 1,00$. The latter estimate describes the (worst) case where after N observations $y(j)$ of the reference material y_R being encompassed by the interval $[y_R - U_p;\ y_R + U_p]$, the next (future) observation y' of the same reference material is not encompassed by that interval. This demonstrates the underestimating character of the robust estimate $p = M/(N + 1)$.

The standard error of the estimate $p = M/(N + 1)$ can be described by $s(p) = \sqrt{p(1-p)/(N+1)}$. A lower 95 % limit p_L for the true coverage probability π is given by $p_L = p - 1{,}64 \cdot s(p)$ for $N \geqslant 20$ [10]. In this case, the risk of the true coverage probability π being smaller than p_L is $\alpha = 5$ %. Equivalently, the so-called type I error of stating $\pi \geqslant p_L$ is $\alpha = 5$ %. The lower 95 % limit p_L can be called as well a lower 95 % confidence limit for the true coverage probability π.

These considerations apply as well, if more than a single reference material or a reference method are used to provide artefacts of the measurand. In these cases, the reference value y_R is replaced by $y_R(j)$.

Table A.1 shows examples of robust coverage probability $p = M/(N + 1)$, the corresponding standard error $s(p)$ and the lower 95 % limit p_L.

Table A.1 — Robust estimation of coverage probability p

N	M	$p = M/(N + 1)$	$s(p)$	$p_L = p - 1{,}64 \cdot s(p)$
20	20	0,95	0,046	0,88
20	19	0,90	0,064	0,80
40	40	0,98	0,024	0,94
40	39	0,95	0,034	0,90
60	60	0,98	0,016	0,96
60	59	0,97	0,023	0,93
60	58	0,95	0,028	0,91
80	80	0,99	0,012	0,97
80	79	0,98	0,017	0,95
80	78	0,96	0,021	0,93
80	77	0,95	0,024	0,91
100	100	0,99	0,010	0,97
100	99	0,98	0,014	0,96
100	98	0,97	0,017	0,94
100	97	0,96	0,019	0,93
100	96	0,95	0,022	0,92
200	191	0,95	0,015	0,92

EXAMPLE 1 For finding all of $N = 20$ observations of a reference material y_R encompassed by the uncertainty interval $[y_R(1 - \varDelta);\ y_R(1 + \varDelta)]$ provided by expanded uncertainty $U_p = \varDelta \cdot y$, a robust estimate of coverage probability is given by $p = N/(N + 1) = 0{,}95$ with standard error $s(p) = 0{,}046$. The lower 95 % limit for the true coverage probability is given by $p_L = 0{,}88$.

EXAMPLE 2 For finding $M = 39$ of $N = 40$ observations of a reference material y_R encompassed by the uncertainty interval $[y_R(1 - \varDelta);\ y_R(1 + \varDelta)]$ provided by expanded uncertainty $U_p = \varDelta \cdot y$, a robust estimate of coverage probability is given by $p = M/(N + 1) = 0{,}95$ with standard error $s(p) = 0{,}034$. The lower 95 % limit for the true coverage probability is given by $p_L = 0{,}90$. In this case, the expanded 95 % uncertainty $U_{0,95} = \varDelta \cdot y$ is expected to ensure a coverage probability of at least 90 % for uncertainty intervals $[y'(1 - \varDelta);\ y'(1 + \varDelta)]$ about future observations y'.

EXAMPLE 3 For finding all of $N = 60$ observations of a reference material y_R encompassed by the uncertainty interval $[y_R(1-\Delta); y_R(1+\Delta)]$ provided by expanded uncertainty $U_p = \Delta \cdot y$, a robust estimate of coverage probability is given by $p = N/(N+1) = 0{,}98$ with standard error $s(p) = 0{,}016$. The lower 95 % limit for the true coverage probability is given by $p_L = 0{,}96$.

A.3 Testing a coverage probability

In the following, a test of a coverage probability p assigned to a given value of the expanded uncertainty $U_p = k \cdot u$ or $U_p = \Delta \cdot y$ is illustrated. For the sake of simplicity, a series of N observations $y(j)$ of a reference material of certified value y_R is considered.

The probability or risk α of finding less than M in N observations $y(j)$ of a reference material y_R fulfilling the relationship $y_R - U_p \leqslant y(j) \leqslant y_R + U_p$ is determined by Equation (A.1) [10]:

$$\alpha = 1 - \sum_{k=M}^{N} \binom{N}{k} \cdot p^k \cdot (1-p)^{N-k} \tag{A.1}$$

where p is the true value of the assigned coverage probability.

Table A.2 summarizes typical values of risk α for a coverage probability $p = 0{,}95$. Table A.2 applies as well, if more than a single reference material or a reference method are used as artefacts of the measurand. In these cases, the reference value y_R is replaced by $y_R(j)$.

Table A.2 — Risk α of finding less than M of N observations $y(j)$ fulfilling relationship $y_R - U_p \leqslant y(j) \leqslant y_R + U_p$ for a coverage probability $p = 0{,}95$

M	α					
	$N = 20$	$N = 40$	$N = 60$	$N = 80$	$N = 100$	$N = 200$
N	0,64	0,87	0,95	0,98	0,99	1,00
$N-1$	0,26	0,60	0,81	0,91	0,96	1,00
$N-2$	0,08	0,32	0,58	0,77	0,88	1,00
$N-3$	0,02	0,14	0,35	0,57	0,74	0,99
$N-4$	0,00	0,05	0,18	0,37	0,56	0,97
$N-5$	0,00	0,01	0,08	0,21	0,38	0,94
$N-6$	0,00	0,00	0,03	0,11	0,23	0,88
$N-7$	0,00	0,00	0,01	0,05	0,13	0,79
$N-8$	0,00	0,00	0,00	0,02	0,06	0,67
$N-9$	0,00	0,00	0,00	0,01	0,03	0,55
$N-10$	0,00	0,00	0,00	0,00	0,01	0,42
$N-15$	0,00	0,00	0,00	0,00	0,00	0,04

EXAMPLE 1 According to Table A.2, the risk α of finding less than $M = 19$ of $N = 20$ (future) observations $y(j)$ fulfilling the relationship $y_R - U_{0,95} \leqslant y(j) \leqslant y_R + U_{0,95}$ for a given value of the expanded uncertainty $U_{0,95} = 2 \cdot u$ is given by $\alpha = 26$ %. Finding less then $M = 17$ in $N = 20$ (future) observations fulfilling the relationship is very unlikely ($\alpha = 0{,}02$) and could be a reason to reject the estimate $U_{0,95} = 2 \cdot u$ with a type I error smaller than 5 %. In the latter case, a coverage probability $p < 0{,}95$ should be assigned to $U_{0,95} = 2 \cdot u$.

EXAMPLE 2 According to Table A.2, the risk α of finding less than M = 39 of N = 40 (future) observations $y(j)$ fulfilling the relationship $y_R - U_{0,95} \leqslant y(j) \leqslant y_R + U_{0,95}$ for a given value of the expanded uncertainty $U_{0,95} = 2 \cdot u$ is given by α = 0,32. Finding less then M = 36 in N = 40 (future) observations fulfilling the relationship is very unlikely (α = 0,05) and could be a reason to reject the estimate $U_{0,95} = 2 \cdot u$ with a type I error of 5 %. In the latter case, a coverage probability p < 0,95 should be assigned to $U_{0,95} = 2 \cdot u_c$.

EXAMPLE 3 To find all of N = 60 (future) observations $y(j)$ in the uncertainty interval $[y_R(1 - \Delta); y_R(1 + \Delta)]$ for a given data quality objective Δ is quite unlikely ($1 - \alpha$ = 0,05) with a coverage probability p = 0,95. In fact, it could be a reason to reject hypothesis p = 0,95 with a type I error of 5 % in favour of $p > 0{,}95$.

Annex B
(informative)

Type A evaluation methods for experimental designs A1 to A8

B.1 General

This annex provides Type A evaluation methods for the experimental designs A1 to A8 considered in this International Standard (see 5.4). Table B.1 provides an overview. The description of these methods in B.2 to B.10 comprises comprehensive information on the mathematical procedure and a scheme for numerical evaluation.

Table B.1 — Overview on Type A evaluation methods

Type	Description	Clause
A1	Simple random sampling	B.2
A2	Observation of a reference material by a measuring system	B.3
A3	Observation of reference materials in a calibration procedure	B.4
A4	Observation of reference materials by identical measuring systems	B.5
A5, case 1	Parallel measurements with a reference method in a calibration procedure	B.6
A5, case 2	Parallel measurements with a reference method in an evaluation procedure	B.7
A6	Paired measurements with identical measuring systems	B.8
A7	Interlaboratory comparison of identical measuring systems	B.9
A8	Parallel measurements with identical measuring systems	B.10

Each of the described Type A evaluation methods can be applied either in a direct approach or as part of an indirect approach. In a direct approach, the evaluated quantity is the result of measurement y. In an indirect approach, the evaluated quantity is an input quantity x_i of the applicable analytical equation $y = f(x_1,..., x_K)$ used to calculate the result of measurement. For simplicity, the result of measurement y is treated in B.2 to B.10 as the evaluated quantity. If these evaluation methods are applied as part of an indirect approach, the input quantity x_i becomes the evaluated quantity y for this sub-evaluation.

The basic elements and instructions for application of the evaluation procedures A1 to A8 are provided in tabular form. These tables also specify the information to be provided by the user during application of the corresponding evaluation method.

Tables B.2 to B.10 can be used as templates for specific applications. In this case, they have to be amended by information specific to the application considered (see examples in Annex C).

If appropriate, an analytical equation used to calculate the quantity to be evaluated is specified in the tables.

If appropriate, notes are amended to provide additional information (e.g. modifications applicable in the case of signal proportional uncertainties).

B.2 Simple random sampling

Table B.2 specifies the evaluation method, which applies to experimental design A1 (simple random sampling). Examples of evaluation method A1 are given in the GUM.

Table B.2 — Evaluation method for experimental design A1

Step	Element	Instruction
1	**Problem specification**	
	Evaluated quantity	Result of measurement y
	Input data	Series of unbiased observations $y(j)$ with j = 1 to N of the same measurand using the same measuring system
	Reference value	Mean value $\bar{y} = \frac{1}{N} \sum_{j=1}^{N} y(j)$
	Additional information	Reference value $\bar{y}$ is treated as an unbiased estimate of the wanted true value of quantity $y(j)$
2	**Statistical analysis**	
	Data model	$y(j) = \bar{y} + e(j)$ with $e(j) = y(j) - \bar{y}$
	Variance equation	$\mathrm{var}(y) = \frac{1}{N}\mathrm{var}(y) + u^2(e) + 2 \cdot \mathrm{cov}(\bar{y}, e)$
	Residual standard deviation	$u(e) = \sqrt{\frac{1}{N}\sum_{j=1}^{N}\left(y(j) - \bar{y}\right)^2}$
	Covariance	$\mathrm{cov}(\bar{y}, e) = 0$
	Bias of $y(j)$	Necessary *a priori* knowledge: $y(j)$ = 0
3	**Evaluation of uncertainty parameters**	
	Standard uncertainty	$u(y) = s(y) = \sqrt{\frac{1}{N-1}\sum_{j=1}^{N}\left(y(j) - \bar{y}\right)^2}$
	Number of degrees of freedom	If the set of input data $y(j)$ with j = 1 to N is unbiased, $\nu = N - 1$
	Range of application	$\min(y) \leqslant y \leqslant \max(y)$

Type A1 evaluation method can be applied only, if the series of input data $y(j)$ with j = 1 to N is known in advance to be unbiased or at least to exhibit a negligible bias. In general, this requires *a priori* demonstration of quantity y to be unbiased.

B.3 Repeated observation of a reference material by a measuring system

Table B.3 specifies the evaluation method, which applies to experimental design A2 (repeated observation of a reference material by a measuring system). An example of evaluation method A2 is given in C.3.

Table B.3 — Evaluation method for experimental design A2

Step	Element	Instruction
1	**Problem specification**	
	Evaluated quantity	Result of measurement y
	Input data	Series of observations $y(j)$ with j = 1 to N of a reference material using the same measuring system
	Reference value	Accepted value of the reference material y_R
	Additional information	Standard uncertainty of reference material $u(y_R)$
2	**Statistical analysis**	
	Data model	$y(j) = y_R + e(j)$ with residual deviation $e(j) = y(j) - y_R$
	Variance equation	$\text{var}(y) = u^2(y_R) + u^2(e) + 2 \cdot \text{cov}(y_R, e)$
	Residual standard deviation	$u(e) = \sqrt{\frac{1}{N}\sum_{j=1}^{N}(y(j) - y_R)^2}$
	Covariance	$\text{cov}(y_R, e) = 0$
	Bias	$u_B = \left\lvert \bar{y} - y_R \right\rvert = \left\lvert \frac{1}{N}\sum_{j=1}^{N} y(j) - y_R \right\rvert$
3	**Evaluation of uncertainty parameters**	
	Standard uncertainty	$u(y) = \sqrt{u^2(y_R) + u^2(e)}$
	Number of degrees of freedom	If $u(y) \cong u(e)$, then $\nu = N$. Otherwise, modify the procedure used to provide input data.
	Range of application	$\min(y) \leqslant y \leqslant \max(y)$

The standard uncertainty $u(y)$ can be calculated equivalently by Equation (B.1):

$$u(y) = \sqrt{s^2(y)\left(1 - \frac{1}{N}\right) + u_B^2 + u^2(y_R)} \tag{B.1}$$

where $s(y)$ is the standard deviation of the input data $y(j)$ with j = 1 to N:

$$s(y) = \sqrt{\sum_{j=1}^{N} \frac{(y(j) - \bar{y})^2}{N - 1}}$$

B.4 Observation of different reference materials in a calibration procedure

Table B.4 specifies the elements of uncertainty estimation, which applies to experimental design A3 (observation of different reference materials in a calibration procedure). An example of evaluation method A3 is given in C.4.

Table B.4 — Elements of uncertainty estimation for experimental design A3

Step	Element	Instruction
1	**Problem specification**	
	Evaluated quantity	Result of measurement y
	Analytical function	$y = x / b$ where x is the uncorrected response of the measuring system; b is the correcting factor.
	Input data	Series of observations $x(j)$ with j = 1 to N of K reference materials in a calibration procedure with $K < N$
	Reference values	Series of reference values $y_R(j)$ with $j = 1$ to N with value $y_R(j)$ assigned to the reference material described by observed value $x(j)$ According to the number M of repeated observations of a reference material, the same value is assigned M-times to the set of reference values $y_R(j)$ with $j = 1$ to N
	Additional information	Constant standard uncertainty of the reference material $u(y_R(j)) = u(y_R)$ Assumption of constant standard uncertainty $u(y)$
2	**Statistical analysis**	
	Method model equation	$y = x / b$
	Variance equation	$\mathrm{var}(y) = \left(\frac{u(x)}{b}\right)^2 + \left(\frac{y \cdot u(b)}{b}\right)^2 - 2 \cdot \mathrm{cov}(x,b)$
	Covariance	$\mathrm{cov}(x,b) = 0$
	Bias	Zero due to correction by means of analytical equation
3	**Evaluation of input quantities**	
	Model equation of calibration	$x(j) = b \cdot y_R(j) + e_x(j)$ with residual deviation $e_x(j) = x(j) - b \cdot y_R(j)$
	Correction factor	$b = \frac{\bar{x}}{\bar{y}_R} = \left(\sum_{j=1}^{N} x(j)\right) / \left(\sum_{j=1}^{N} y_R(j)\right)$
	Residual standard deviation	$u(e_x) = \sqrt{\frac{1}{N-1} \sum_{j=1}^{N} \left(x(j) - b \cdot y_R(j)\right)^2}$
	Standard uncertainty of quantity x	$u(x) = u(e_x)$
	Standard uncertainty of correction factor b	$u(b) = b \cdot \sqrt{\frac{1}{N}\left(\frac{u(x)}{\bar{x}}\right)^2 + \frac{1}{K}\left(\frac{u(y_R)}{\bar{y}_R}\right)^2}$
4	**Evaluation of uncertainty parameters**	
	Standard uncertainty	$u(y) = \sqrt{\left(\frac{u(e_x)}{b}\right)^2 + y^2 \left(\frac{u(b)}{b}\right)^2}$
	Number of degrees of freedom	If $u(y) \cong u(e_x) / b$, then $\nu = N - 1$. Otherwise, see 7.4.
	Range of application	$\min(y) \leqslant y \leqslant \max(y)$

Method A3 can be used to evaluate a calibration procedure executed separately to a single measuring system of a specified type. In this case, the series of N observations to be evaluated is provided by repeated (M-times) observation of each of $K = N / M$ different reference materials using the same measuring system.

Method A3 can be applied to a method validation procedure performed with the aim to provide bias-correction applicable to all measuring systems of a specified type. In this case, the series of N observations to be evaluated is provided by parallel exposition of M sampling systems of the specified make to $K = N / M$ different test-gas atmospheres.

If appropriate, the applied regression technique [11] can be replaced by any other documented regression technique providing an estimate of slope b and its standard uncertainty $u(b)$.

The case of constant relative uncertainty $u(y)/y$ is treated in evaluation method A4.

B.5 Observation of different reference materials by identical measuring systems

Table B.5 specifies the elements of uncertainty estimation, which applies to experimental design A4 (observation of different reference materials by identical measuring systems). An example of evaluation method A4 is given in C.5.

Table B.5 — Elements of uncertainty estimation for experimental design A4

Step	Element	Instruction
1	**Problem specification**	
	Evaluated quantity	Result of measurement y
	Analytical function	$y = x / b$ where x is the uncorrected response of the measuring system; b is the correcting factor.
	Input data	Series of observations $x(j)$ with j = 1 to N of K reference materials in a calibration procedure with $K < N$
	Reference values	Series of reference values $y_R(j)$ with j = 1 to N According to the number M of repeated observations of a reference material, the same value is assigned M-times to the series of reference values $y_R(j)$
	Additional information	Constant relative standard uncertainty of the reference material, $u(y_R)/y_R$ Assumption of constant relative uncertainty $u(y)/y$
2	**Statistical analysis**	
	Method model equation	$y = x / b$
	Variance equation	$\mathrm{var}(y) = \left(\frac{u(x)}{b}\right)^2 + \left(\frac{y \cdot u(b)}{b}\right)^2 - 2 \cdot \mathrm{cov}(x,b)$
	Covariance	$\mathrm{cov}(x,b) = 0$
	Bias of y	Zero due to correction by means of analytical equation
3	**Evaluation of input quantities**	
	Model equation	$x(j) = y_R(j) \cdot (b + e_x(j))$ with relative deviation $e_x(j) = \frac{x(j)}{y_R(j)} - b$
	Correction factor	$b = \frac{1}{N} \sum_{j=1}^{N} \frac{x(j)}{y_R(j)}$
	Residual standard deviation	$u(e_x) = s\left(\frac{x}{y_R}\right) = \sqrt{\frac{1}{N-1} \sum_{j=1}^{N} \left(\frac{x(j)}{y_R(j)} - b\right)^2}$
	Relative standard uncertainty of quantity x	$\frac{u(x)}{x} = \frac{u(e_x)}{b}$
	Standard uncertainty of correction factor b	$u(b) = \frac{u(e_x)}{\sqrt{N}}$
4	**Evaluation of uncertainty parameters**	
	Relative standard uncertainty	$w(y) = \frac{s\left(\frac{x}{y_R}\right)}{b} \cdot \sqrt{\left(1 + \frac{1}{N}\right)}$
	Number of degrees of freedom	If $w(y) \cong \frac{1}{b} \cdot s\left(\frac{x}{y_R}\right)$, then $\nu = N - 1$. Otherwise, see 7.4.
	Range of application	$\min(y) \leqslant y \leqslant \max(y)$

Method A4 can be applied to a method evaluation procedure as performed in industrial hygiene with the aim to provide bias-correction applicable to all measuring systems of a specified type. In this case, the series of N observations to be evaluated is provided by parallel exposition of M sampling systems of the specified make to $K = N / M$ different test-gas atmospheres.

Method A4 can be applied to a calibration procedure executed separately to each measuring system of a specified type. In this case, the series of N observations to be evaluated is provided by repeated (M-times) observation of each of $K = N / M$ different reference materials using the same measuring system.

If appropriate, the applied regression technique [11] can be replaced by any other documented regression technique providing an estimate of slope b and its standard uncertainty $u(b)$.

The case of constant absolute uncertainty $u(y)$ is treated in evaluation method A3.

B.6 Parallel measurements with a reference method of measurement in a calibration procedure

Table B.6 specifies the elements of uncertainty estimation, which applies to experimental design A5 Case 1 (parallel measurements with a reference method of measurement in a calibration procedure). An example of evaluation method A5 Case 1 is given in C.6.

Table B.6 — Elements of uncertainty estimation for experimental design A5 Case 1

Step	Element	Instruction
1	**Problem specification**	
	Evaluated quantity	result of measurement y
	Analytical function	$y = a + b \cdot (x - c)$ where y is the result of measurement; x is the uncorrected response of the measuring system; a, b, c are the parameters of the analytical function.
	Input data	Series of uncorrected response values $x(j)$ with j = 1 to N of the measuring system obtained in parallel measurements with the reference method providing results $y_{\mathrm{R}}(j)$ in a calibration procedure
	Reference values	Results of measurement $y_{\mathrm{R}}(j)$ with j = 1 to N obtained by the reference method in the calibration procedure
	Additional information	Assumption of constant standard uncertainty of the reference method $u(y_{\mathrm{R}})$ Assumption of constant uncertainty $u(y)$
2	**Statistical analysis**	
	Statistical model equation	$y = a + b \cdot (x - c) + e_y$
	Variance equation	$\mathrm{var}(y) = u^2(a) + u^2(b) \cdot (x - c)^2 + b^2 \cdot u^2(c) + u^2(e_y)$
	Covariance	$\mathrm{cov}(x,a) = \mathrm{cov}(x,b) = \mathrm{cov}(x,c) = 0$ $\mathrm{cov}(a,b) = \mathrm{cov}(b,c) = \mathrm{cov}(c,a) = 0$
3	**Evaluation of input quantities**	
	Model equation of calibration	$y_{\mathrm{R}}(j) = a + b \cdot (x(j) - c) + e_y(j)$ with deviation $e_y(j) = y_{\mathrm{R}}(j) - a - b \cdot (x(j) - c)$
	Parameter a	$a = \bar{y}_{\mathrm{R}} = \frac{1}{N} \sum_{j=1}^{N} y_{\mathrm{R}}(j)$
	Parameter b	$b = \frac{\sum_{j=1}^{N} (y_{\mathrm{R}}(j) - a) \cdot (x(j) - c)}{\sum_{j=1}^{N} (x(j) - c)^2}$
	Standard uncertainty of b	$u(b) \cong \sqrt{\frac{u^2(e_y)}{\sum_{j=1}^{N} (x(j) - c)^2}}$
	Parameter c	$c = \bar{x} = \frac{1}{N} \sum_{j=1}^{N} x(j)$
	Residual standard deviation	$u(e_y) = \sqrt{\frac{1}{N-2} \sum_{j=1}^{N} e_y^2(j)}$
4	**Evaluation of uncertainty parameters**	
	Standard uncertainty	$u(y) = \sqrt{\left(1 + \frac{1}{N}\right) \cdot u^2(e_y) + \left(\frac{u(b)}{b}\right)^2 (y - \bar{y}_{\mathrm{R}})^2}$
	Number of degrees of freedom	If $u(y) \cong u(e_y)$, then $\nu = N - 2$. Otherwise, see 7.4.
	Range of application	$\min(y) \leqslant y \leqslant \max(y)$

The uncertainty $u(y_R)$ of the reference method and of the response values x are taken into account implicitly by $u(e_y)$.

In the case of signal proportional uncertainties with $u(y)/y$ = constant and $u(y_R)/y_R$ = constant and for $N \geqslant 10$, the relative standard uncertainty $w(y)$ of the evaluated quantity y is estimated in good approximation by Equation (B.2), which is applicable under the above mentioned conditions only:

$$w(y) = \frac{u(y)}{y} \cong \sqrt{\left(1+\frac{1}{N}\right)\cdot\frac{1}{N-2}\sum_{j=1}^{N}\left(\frac{y(j)}{y_R(j)}-1\right)^2 + \left(\frac{u(b)}{b}\right)^2\cdot\left(\frac{y-a}{y}\right)^2} \qquad \text{(B.2)}$$

The analytical equation $y = a + b \cdot (x - c)$ is equivalent to the equation $y = A + b \cdot x$ with $A = a - b \cdot c$.

If appropriate, the applied ordinary least square regression technique [11] can be replaced by any other documented regression technique, providing an estimate of slope b and its standard uncertainty $u(b)$.

B.7 Parallel measurements with a reference method of measurement in an evaluation procedure

Table B.7 specifies the elements of uncertainty estimation, which applies to experimental design A5 Case 2 (parallel measurements with a reference method of measurement in an evaluation procedure). An example of evaluation method A5 Case 2 is given in C.7.

Table B.7 — Elements of uncertainty estimation for experimental design A5 Case 2

Step	Element	Instruction
1	**Problem specification**	
	Evaluated quantity	Result of measurement y
	Input data	Series of observations $y(j)$ with j = 1 to N provided by the measuring system obtained in parallel with reference method results $y_R(j)$ in an evaluation procedure.
	Reference values	Series of observations $y_R(j)$ obtained by the reference method applied in parallel with the measuring system.
	Additional information	Input data are not used to correct the measuring system; constant standard uncertainty $u(y_R)$ of the reference method; assumption of constant uncertainty $u(y)$.
2	**Statistical analysis**	
	Statistical model equation	$y(j) = y_R(j) + e_y(j)$ with deviation $e_y(j) = y(j) - y_R(j)$
	Variance equation	$\text{var}(y) = u^2(y_R) + u^2(e_y) + 2 \cdot \text{cov}(y_R, e_y)$
	Covariance	$\text{cov}(y_R, e_y) = -u^2(y_R)$
	Residual standard deviation	$u(e_y) = \sqrt{\frac{1}{N}\sum_{j=1}^{N}(y(j) - y_R(j))^2}$
	Bias	$u_B(y) = \frac{1}{N}\sum_{j=1}^{N}(y(j) - y_R(j))$
3	**Evaluation of uncertainty parameters**	
	Standard uncertainty	$u(y) = \sqrt{\frac{1}{N}\sum_{j=1}^{N}(y(j) - y_R(j))^2 - u^2(y_R)}$
	Number of degrees of freedom	If $u_B^2(y) \leqslant 0{,}5 \cdot u^2(y)$, then $\nu = N$.
	Range of application	$\min(y) \leqslant y \leqslant \max(y)$

The equation for estimating $u(y)$ is applicable only, if the standard uncertainty of the reference method $u(y_R)$ is obtained by a Type A evaluation and fulfils the relationship $u(y_R) \leqslant 0{,}3 \cdot u(y)$. Otherwise, $u(y_R)$ is replaced by zero in order to obtain a conservative estimate of $u(y)$.

In the case of signal proportional uncertainties with $u(y)/y$ = constant and $u(y_R)/y_R$ = constant and for $N \geqslant 10$, the relative standard uncertainty $w(y)$ of the evaluated quantity y is estimated in good approximation by Equation (B.3), which is applicable under the above mentioned conditions only:

$$w(y) = \frac{u(y)}{y} = \sqrt{\frac{1}{N}\sum_{j=1}^{N}\left(\frac{y(j)}{y_R(j)} - 1\right)^2 - \left(\frac{u(y_R)}{y_R}\right)^2} \qquad \text{(B.3)}$$

B.8 Paired measurements of two identical measuring systems

Table B.8 specifies the elements of uncertainty estimation, which applies to experimental design A6 (paired measurements of two identical measuring systems). An example of evaluation method A6 is given in C.8.

Table B.8 — Elements of uncertainty estimation for experimental design A6

Step	Element	Instruction
1	**Problem specification**	
	Evaluated quantity	Result of measurement y
	Input data	Series of observations $y(1,j)$ and $y(2,j)$ with j = 1 to N obtained in paired application of two identical measuring systems operated independently of each other
	Reference values	Mean values $y_R(j) = (y(1,j) + y(2,j))/2$
	Additional information	Constant standard uncertainty of the reference method, $u(y_R) = u(y)/\sqrt{2}$; assumption of constant uncertainty $u(y)$.
2	**Statistical analysis**	
	Statistical model equation	$y(1,j) = y_R(j) + e(1,j)$ $y(2,j) = y_R(j) + e(2,j)$ with deviations $e(1,j) = \left(y(1,j) - y(2,j)\right)/2$ $e(2,j) = -e(1,j)$
	Variance equation	$\mathrm{var}\left(y(1,j)\right) = \mathrm{var}\left(y(1,j)\right)/2 + \frac{1}{4N}\sum_{j=1}^{N}\left(y(1,j) - y(2,j)\right)^2$ $\mathrm{var}\left(y(2,j)\right) = \mathrm{var}\left(y(1,j)\right)$
	Covariance	$\mathrm{cov}\left(y_R(j), e(k,j)\right) = 0$
	Bias	$u_B(y) = \frac{1}{N}\sum_{j=1}^{N}\left(y(1,j) - y(2,j)\right)$
3	**Evaluation of uncertainty parameters**	
	Standard uncertainty	$u(y) = \sqrt{\frac{1}{2 \cdot N}\sum_{j=1}^{N}\left(y(1,j) - y(2,j)\right)^2}$
	Number of degrees of freedom	If $u_B^2(y) \leqslant 0{,}5 \cdot u^2(y)$, then $\nu = N$. Otherwise, see 7.4.
	Range of application	$\min(y) \leqslant y \leqslant \max(y)$

Method A6 is not suited to take into account a common bias of both measuring systems in uncertainty estimation.

In the case of signal proportional uncertainties with $u(y)/y$ = constant, the relative standard uncertainty $w(y)$ of the evaluated quantity y is estimated by Equation (B.4):

$$w(y) = \frac{u(y)}{y} = \sqrt{\frac{1}{2N}\sum_{j=1}^{N}\left(\frac{y(1,j)}{y(2,j)} - 1\right)^2} \tag{B.4}$$

B.9 Interlaboratory comparison of identical measuring systems

Table B.9 specifies the elements of uncertainty estimation, which applies to experimental design A7 (interlaboratory comparison of identical measuring systems). An example of evaluation method A7 is given in C.9.

Table B.9 — Elements of uncertainty estimation for experimental design A7

Step	Element	Instruction
1	**Problem specification**	
	Evaluated quantity	Result of measurement y
	Input data	Series of observations $y(k, j)$ with j = 1 to N of the same (unknown) measurand by identical measuring systems k = 1 to K
	Reference values	Mean value $\bar{\bar{y}} = \frac{1}{K \cdot N} \sum_{k=1}^{K} \sum_{j=1}^{N} y(k,j)$
	Additional information	Assumption of constant uncertainty $u(y)$. Reference value $\bar{\bar{y}}$ is taken as an unbiased estimate of the true value of the measurand.
2	**Statistical analysis**	
	Data model	$y(k,j) = \bar{\bar{y}} + a(k) + e(k,j)$ with residual deviation $e(k,j) = y(k,j) - \overline{y(k)}$, laboratory bias $a(k) = \overline{y(k)} - \bar{\bar{y}}$, laboratory mean $\overline{y(k)} = \frac{1}{N} \sum_{j=1}^{N} y(k,j)$
	Variance equation	$\mathrm{var}(y) = u^2(\bar{\bar{y}}) + u^2(a) + u^2(e)$
	Covariance	$\mathrm{cov}(\bar{\bar{y}},e) = \mathrm{cov}(a,e) = \mathrm{cov}(\bar{\bar{y}},a) = 0$
3	**Evaluation of uncertainty parameters**	
	Repeatability standard deviation	$u(e) = s_r(y) = \sqrt{\frac{1}{K} \sum_{k=1}^{K} s^2(k)}$
	Laboratory standard deviation	$s(k) = \sqrt{\frac{1}{N-1} \sum_{j=1}^{N} \left(y(k,j) - \overline{y(k)}\right)^2}$
	Interlaboratory variation	$u(a) = \sqrt{\frac{1}{K} \sum_{k=1}^{K} \left(\overline{y(k)} - \bar{\bar{y}}\right)^2}$
	Standard uncertainty of reference value	$u(\bar{\bar{y}}) = \sqrt{\frac{1}{K} u^2(a)}$
	Standard uncertainty	$u(y) = \sqrt{\frac{1}{K-1} \sum_{k=1}^{K} \left(\overline{y(k)} - \bar{\bar{y}}\right)^2 + s_r^2(y)}$
	Number of degrees of freedom	If $u(y) \cong u(a)$, then $\nu \cong K - 1$ If $u^2(a) \leqslant 0{,}5 \cdot u^2(y)$, then $\nu \cong K \cdot N - 1$ Otherwise, see 7.4.
	Range of application	$\min(y) \leqslant y \leqslant \max(y)$

Method A7 is not suited to take into account a common bias of the compared measuring systems in uncertainty estimation.

B.10 Parallel application of identical measuring systems under field conditions

Table B.10 specifies the elements of uncertainty estimation, which applies to experimental design A8 (parallel application of identical measuring systems under field conditions). An example of evaluation method A8 is given in C.10.

Table B.10 — Elements of uncertainty estimation for experimental design A8

Step	Element	Instruction
1	**Problem specification**	
	Evaluated quantity	Result of measurement y
	Input data	Series of observations $y(k, j)$ obtained in trial j = 1 to N by measuring systems k = 1 to K
	Reference values	Mean value $y_\mathrm{R}(j) = \frac{1}{K}\sum_{k=1}^{K} y(k,j)$ for trial j
	Additional information	Assumption of constant uncertainty $u(y)$. Reference values are taken as unbiased estimates for each trial.
2	**Statistical analysis**	
	Data model	$y(k,j) = y_\mathrm{R}(j) + e(k,j)$ with residual deviation $e(k,j) = y(k,j) - y_\mathrm{R}(j)$
	Variance equation	$\mathrm{var}(y) = \frac{1}{N}\sum_{j=1}^{N} s^2(j)$
	Standard deviation in each trial	$s(j) = \sqrt{\frac{1}{K-1}\sum_{k=1}^{K}\left(y(k,j) - y_\mathrm{R}(j)\right)^2}$
	Covariance	$\mathrm{cov}\left(y_\mathrm{R}(j), e(k,j)\right) = 0$
	Instrument bias	$a(k) = \overline{y_k} - \overline{\overline{y}}$ with $\overline{y_k} = \frac{1}{N}\sum_{j=1}^{N} y(k,j)$ and $\overline{\overline{y}} = \sum_{k=1}^{K}\overline{y_k}$
	Bias	$u_\mathrm{B}(y) = \sqrt{\frac{1}{K}\sum_{k=1}^{K} a^2(k)}$
3	**Evaluation of uncertainty parameters**	
	Standard uncertainty	$u(y) = \sqrt{\frac{1}{N}\sum_{j=1}^{N} s^2(j)}$
	Number of degrees of freedom	If $u(y) \cong u_\mathrm{B}(y)$, then $\nu \cong K$. If $u_\mathrm{B}^2(y) \leqslant 0{,}5 \cdot u^2(y)$, then $\nu \cong N\,(K-1)$. Otherwise, see 7.4.
	Range of application	$\min(y) \leqslant y \leqslant \max(y)$

Method A8 is not suited to take into account a common bias of the compared measuring systems in uncertainty estimation.

In the case of signal proportional uncertainties with $u(y)/y$ = constant, the relative standard uncertainty $w(y)$ of the evaluated quantity y is estimated by Equation (B.5):

$$w(y) = \frac{u(y)}{y} = \sqrt{\frac{1}{N(K-1)} \sum_{j=1}^{N} \sum_{k=1}^{K} \left(\frac{y(k,j)}{y_{\mathrm{R}}(j)} - 1 \right)^2} \qquad \text{(B.5)}$$

Annex C
(informative)

Examples

C.1 Introduction

This annex comprises examples demonstrating practical application of this International Standard. The work steps and obtained results as well as the evaluated input data are provided for each example in separate tables. These tables can be used as forms to support future applications of the described type.

The main tables in C.3 to C.10 are divided into the three major parts: problem specification, data treatment and uncertainty parameters. Step 2 (data treatment) summarizes the closely linked mathematical work steps statistical analysis and estimation of variances and covariances under a common headline.

Table C.1 provides an overview on the examples.

Table C.1 — Examples

Clause	Example	Experimental design
C.2	Provided by the GUM	A1
C.3	Control of ozone measuring system by means of check-standards	A2
C.4	Calibration of gas-chromatograph using standard solutions of benzene	A3
C.5	Evaluation of toluene measuring method for use in industrial hygiene	A4
C.6	Calibration of an automated emission measuring system	A5 Case 1
C.7	Passive sampling of nitrogen dioxide compared with a reference method	A5 Case 2
C.8	Paired measurements of mercury in stationary source emissions by a manual method of measurement	A6
C.9	Interlaboratory comparison of a measuring method of carbon monoxide in ambient air	A7
C.10	Field-validation of a measuring method of lead in ambient air	A8

C.2 Simple random sampling

Examples of evaluation method A1 are provided by the GUM.

C.3 Control of ozone measuring system by means of check-standards

This example demonstrates the evaluation of a set of input data provided by daily control of an automatic ozone measuring system by means of evaluation method A2 described in Annex B.3.

The work steps and the obtained results are described in Table C.2. Table C.3 comprises the evaluated input data. A comprehensive presentation of resulting uncertainty parameters is given in Table C.4.

The analysis provided the following results. The standard uncertainty $u(y)$ of hourly ozone concentrations y in the interval 10 µg/m³ $\leqslant y \leqslant$ 240 µg/m³ is found within the range 1 µg/m³ $\leqslant u(y) \leqslant$ 8,9 µg/m³. The expanded 95 % uncertainty $U_{0,95}(y)$ stabilizes for 60 µg/m³ $\leqslant y \leqslant$ 240 µg/m³ at 8 %.

The obtained uncertainty parameters are appropriate to characterize the uncertainty of the hourly values of ozone obtained by the controlled measuring system within the evaluated time period of 20 days.

Table C.2 — Work steps and results

Step	Element	Instruction	Result
1	**Problem specification**		
	Method of measurement	Automatic measuring method of ozone in ambient air utilizing UV absorption	—
	Control conditions	Application of zero gas and span gas every 25 h and daily zero-correction; zero and span gas of ozone are produced daily by an ozone generator.	—
	Ambient conditions	Variations in temperature, pressure, humidity and wind speed	—
	Evaluated quantity	Result of measurement: 1 h average of ozone concentration in ambient air at a fixed outdoor location.	y
	Analytical function	$y = x - e(j)$ where y is the result of measurement; x is the uncorrected response of the measuring system; $e(j)$ is the offset-correction for day j.	—
	Uncertainty parameters	Standard uncertainty of ozone concentration in the range of 10 µg/m³ < y < 280 µg/m³ .... Expanded uncertainty of ozone concentration y on a level of confidence of 95 % in the range of 10 µg/m³ < y < 280 µg/m³	$u(y)$ $U_{0,95}(y)$
	Experimental design	Type A2 in a direct approach: Step 1: Daily application of zero gas and determination of offset correction $e(j)$ by means of relationship $e(j) = x_0(j)$. Step 2: Daily application of span gas produced by ozone generator and determination of factor $\beta(j)$ by means of relationship $\beta(j) = x_s(j) / y_s$.	—
	Input data	Series of offset corrections $e(j)$ with j = 1 to N = 20. Series of observed span factors $\beta(j)$ with j =1 to N = 20.	See Table C.3
	Reference values	Zero-gas y_0 .... Span gas concentration y_s	0 µg/m³ 280 µg/m³
	Additional Information	Standard uncertainty of the zero gas $u(y_0)$ .... Standard uncertainty of the span gas $u(y_s)$ .... Standard uncertainty $u(y)$ is expected to grow in proportion with y.	not specified 2,8 µg/m³

Table C.2 (*continued*)

Step	Element	Instruction	Result
	Representativeness	The evaluated set of input data represents variations in ambient conditions and operating conditions that occurred within the time period of 20 days considered for this example.	—
	Effects not addressed	Influence of the sampling system	—
2	**Data treatment**		
	Model equation	$y = x - e(j)$ with daily zero-correction $e(j) = x_0(j)$	—
	Variance equation	$\text{var}(y) = u^2(x) + u^2(e) + 2 \cdot \text{cov}(x,e)$	—
	Covariance	$\text{cov}(x,e)$	0
	Standard uncertainty of zero-correction e	$u(e) = \sqrt{\frac{1}{N}\sum_{j=1}^{N} x_0^2(j)}$ takes implicitly into account bias $u_B(e)$.	0,89 µg/m³
	Bias of $y(j)$	$u_B(e) = \frac{1}{N}\sum_{j=1}^{N} x_0(j)$	–0,86 µg/m³
	Model equation for $x(j)$	$x(j) = \beta(j) \cdot y_S$	—
	Variance equation	$\text{var}(x) = x^2 \cdot \left[\left(\frac{u(\beta)}{\beta}\right)^2 + \left(\frac{u(y_S)}{y_S}\right)^2\right]$ $+2 \cdot x(j) \cdot \text{cov}(\beta, y_S)$	—
	Covariance	$\text{cov}(\beta, y_S)$	0
	Standard uncertainty of β	$u(\beta) = \sqrt{\frac{1}{N}\sum_{j=1}^{N}(1-\beta(j))^2}$ takes into account implicitly bias $u_B(\beta)$	0,036
	Bias of $\beta(j)$	$u_B(\beta) = \frac{1}{N}\sum_{j=1}^{N}\beta(j) - 1$	0,02
3	**Results of uncertainty analysis**		
	Standard uncertainty of y	$u(y) \cong \sqrt{y^2 \cdot \left[\left(\frac{u(\beta)}{\beta}\right)^2 + \left(\frac{u(y_S)}{y_S}\right)^2\right] + u^2(e)}$	1,0 µg/m³ to 8,9 µg/m³ See Table C.4.
	Number of degrees of freedom	ν since $u(y_S) \leqslant 0{,}5 \cdot u(y)$	20
	Coverage factor	$k_{0,95}$	2,1
	(relative) expanded uncertainty of y	$W_{0,95}(y) = k_{0,95} \cdot u(y) / y$	20 % to 8 % See Table C.4.
	Range of application	$\min(y) \leqslant y \leqslant \max(y)$	10 µg/m³ $\leqslant y \leqslant$ 240 µg/m³

Table C.3 — Input data

Index j	**Zero response** $e(j)$ µg/m³	**Span factor** $\beta(j)$
1	−0,7	1,00
2	−0,9	0,96
3	−1,4	0,98
4	−0,9	0,99
5	−1,1	1,04
6	−0,3	1,05
7	−0,8	1,04
8	−0,8	1,03
9	−1,0	1,04
10	−1,0	1,03
11	−0,9	1,02
12	−0,8	1,02
13	−1,1	1,03
14	−0,8	1,07
15	−0,8	1,04
16	−0,6	1,02
17	−0,5	1,02
18	−1,0	1,05
19	−0,7	1,05
20	−1,0	0,97

Table C.4 — Uncertainty parameters

Result of measurement y µg/m³	Standard uncertainty $u(y)$ µg/m³	Expanded uncertainty $W_{0,95}(y)$ %
10	1,0	20
20	1,2	12
40	1,7	9
60	2,4	8
80	3,1	8
100	3,8	8
120	4,5	8
140	5,2	8
160	5,9	8
180	6,7	8
200	7,4	8
220	8,1	8
240	8,9	8

C.4 Calibration of gas-chromatograph using standard solutions of benzene

This example demonstrates evaluation of a set of input data provided by calibration of a gas chromatograph using standard solutions of benzene in CS_2. Data treatment was performed by means of evaluation method A3 provided in Annex B.4.

The work steps and the obtained results are summarized in Table C.5. The evaluated input data and the obtained calibration line are shown in Table C.6. The resulting uncertainty parameters are presented Table C.7.

The analysis provided the following results. The standard uncertainty $u(y)$ of an individual benzene mass fraction y between 3 µg/g and 16 µg/g is found in the range 0,23 µg/g $< u(y) <$ 0,24 µg/g. The effective number of degrees of freedom is given by $\nu = 28$. The expanded uncertainty on a level of confidence of 95 % for mass fractions y between 3 µg/g and 16 µg/g is found in the range 0,46 µg/g $< U_{0,95}(y) <$ 0,48 µg/g.

Figure C.1 and Figure C.2 provide a graphical impression of the calibration line and the analytical function established by evaluation of the input data.

This evaluation is used to predict the uncertainty of future results of measurement y that are obtained by the calibrated gas chromatograph until next recalibration (e.g. after three years).

Table C.5 — Work steps and results

Step	Element	Instruction	Result
1	**Problem specification**		
	Method of measurement	Pumped sampling of aromatic hydrocarbons on charcoal tube; desorption of benzene by means of CS_2; analytical quantification by gas chromatography, FID	—
	Control conditions	Calibration of the gas chromatograph using standard solutions of benzene in CS_2 every 3 years.	—
	Ambient conditions	Variation in temperature, pressure, humidity and wind speed	—
	Evaluated quantity	Result of measurement: 96 h average of benzene mass fraction in ambient air at a fixed outdoor location.	y
	Analytical function	$y = \frac{x}{b}$ where y is the result of measurement; x is the response of the gas chromatograph in units of peak area (AU); b is the correction factor (for bias).	—
	Uncertainty parameters	Standard uncertainty $u(y)$ in the range of benzene mass fraction 3 µg/g < y < 16 µg/g .... Expanded 95 % uncertainty $U_{0,95}(y)$ of benzene mass fraction in the range of 3 µg/g < y < 16 µg/g	$u(y)$ $U_{0,95}(y)$
	Experimental design	Type A3; K = 16 Standard-solutions of benzene in CS_2 were observed using the gas-chromatograph within the specified calibration procedure.	—
	Input data	Series of observations $x(j)$ with j = 1 to N = 29 of K = 16 standard solutions of benzene obtained by gas-chromatography in the calibration procedure.	See Table C.6.
	Reference values	Reference values $y_R(j)$ with j = 1 to N = 29 belonging to the K = 16 standard solutions. According to the number of repeated observations of a standard solution, the same value can appear several times within the series $y_R(j)$ with j = 1 to N = 29.	See Table C.6.
	Additional information	Standard solutions of benzene in CS_2 were prepared by — pumped sampling on a charcoal tube in a test gas atmosphere of well known benzene fraction, and — desorption of benzene in a charcoal tube by means of CS_2.	—
	Additional information	Each test gas atmosphere was prepared by dilution system starting from a certified reference standard (e.g. BCR CRM 562). The 95 % expanded uncertainties of the reference solutions of benzene in CS_2 were reported smaller than 1 %. The standard uncertainty of the reference values is treated as constant, $u(y_R(j)) = u(y_R)$	0,08 µg/g

Table C.5 (*continued*)

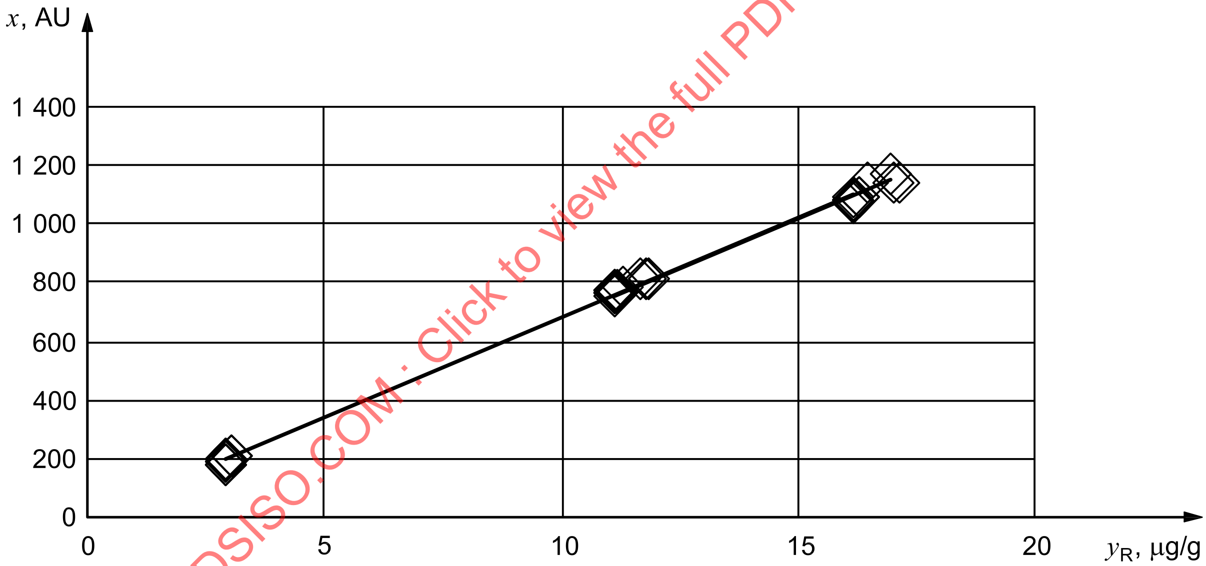
Step	Element	Instruction	Result
	Representativeness	The evaluated calibration procedure is expected to represent sampling, desorption and analytical quantification as described by the applicable method documentation.	—
	Effects not addressed	The data evaluated are not describing the influence of variations in ambient conditions like temperature, humidity and pressure. These effects can require separate considerations.	—
2	**Data treatment**		
	Method model equation	$y = \frac{x}{b}$	—
	Variance equation	$\text{var}(y) = \left(\frac{u(x)}{b}\right)^2 + \left(\frac{y \cdot u(b)}{b}\right)^2 - 2 \cdot \text{cov}(x,b)$	—
	Covariance	$\text{cov}(x,b)$	0
	Bias	Due to correction by means of factor b	0
	Model equation of calibration	$x(j) = b \cdot y_\text{R}(j) + e_x(j)$ with residual deviation $e_x(j) = x(j) - b \cdot y_\text{R}(j)$	—
	Correction factor b	$b = \frac{\bar{x}}{\bar{y}_\text{R}} = \frac{\sum_{j=1}^{N} x(j)}{\sum_{j=1}^{N} y_\text{R}(j)}$	67,92 AU·g/μg
	Residual standard deviation	$u(e_x) = \sqrt{\frac{1}{N-1} \sum_{j=1}^{N} (x(j) - b \cdot y_\text{R}(j))^2}$	14,4 AU
	Standard uncertainty of quantity x	$u(x) = u(e_x)$ .... $u(b)$ and $u(y_\text{R})$ are taken into account separately	14,4 AU
	Standard uncertainty of b	$u(b) = b \cdot \sqrt{\frac{1}{N}\left(\frac{u(x)}{\bar{x}}\right)^2 + \frac{1}{K}\left(\frac{u(y_\text{R})}{y_\text{R}}\right)^2}$	0,28 AU·g/μg
3	**Results of uncertainty analysis**		
	Standard uncertainty of y	$u(y) = \sqrt{\left(\frac{u(e_x)}{b}\right)^2 + y^2 \cdot \left(\frac{u(b)}{b}\right)^2}$	$\geqslant$ 0,21 μg/g See Table C.7.
	Number of degrees of freedom	$\nu = N - 1$ .... since $u(y) \cong u(e_x)$, in good approximation	28
	Coverage factor	$k_{0,95}$	2,05
	Expanded uncertainty of y	$U_{0,95}(y) = k_{0,95} \cdot u(y)$	$\geqslant$ 0,433 μg/g
	Range of application	$\min(y) \leqslant y \leqslant \max(y)$	3 μg/g $\leqslant y \leqslant$ 16 μg/g

Table C.6 — Input data and calibration line

Number	Input data		Calibration line	
	Standard solution	Response	Calibration line	Residuum
j	y_R µg/g	x peak area (AU)	$x' = b\, y_R$ peak area (AU)	e_x peak area (AU)
1	2,891	193,7	196,3	−2,6
2	2,891	182,2	196,3	−14,1
3	2,891	177,7	196,3	−18,6
4	2,891	190,2	196,3	−6,1
5	2,891	194,6	196,3	−1,7
6	2,910	196,0	197,6	−1,6
7	3,035	205,8	206,1	−0,3
8	3,057	205,2	207,6	−2,4
9	11,132	762,1	756,0	6,1
10	11,132	775,1	756,0	19,1
11	11,132	764,7	756,0	8,7
12	11,132	755,8	756,0	−0,2
13	11,132	776,8	756,0	20,8
14	11,204	761,8	760,9	0,9
15	11,204	775,7	760,9	14,8
16	11,310	782,8	768,1	14,7
17	11,684	811,2	793,5	17,7
18	11,771	813,4	799,4	14,0
19	11,841	813,7	804,2	9,5
20	16,190	1095,7	1099,6	−3,9
21	16,190	1085,3	1099,6	−14,3
22	16,190	1084,3	1099,6	−15,3
23	16,190	1068,2	1099,6	−31,4
24	16,295	1091,5	1106,7	−15,2
25	16,295	1094,0	1106,7	−12,7
26	16,448	1141,5	1117,1	24,4
27	16,948	1170,2	1151,0	19,2
28	16,992	1142,3	1154,0	−11,7
29	17,118	1145,2	1162,6	−17,4

Table C.7 — Analytical function and uncertainty interval

x peak area (AU)	$y = x/b$ µg/g	$u(y)$ µg/g	$U_{0,95}(y)$ µg/g	$y - U_{0,95}(y)$ µg/g	$y + U_{0,95}(y)$ µg/g	$U_{0,95}(y)/y$ %
200	2,945	0,227	0,465	2,480	3,410	15,8
300	4,417	0,227	0,466	3,952	4,883	10,5
400	5,890	0,228	0,467	5,423	6,356	7,9
500	7,362	0,229	0,468	6,894	7,830	6,4
600	8,834	0,229	0,470	8,365	9,304	5,3
700	10,307	0,230	0,472	9,835	10,779	4,6
800	11,779	0,232	0,474	11,305	12,254	4,0
900	13,252	0,233	0,477	12,775	13,729	3,6
1000	14,724	0,234	0,480	14,244	15,204	3,3
1100	16,197	0,236	0,483	15,713	16,680	3,0

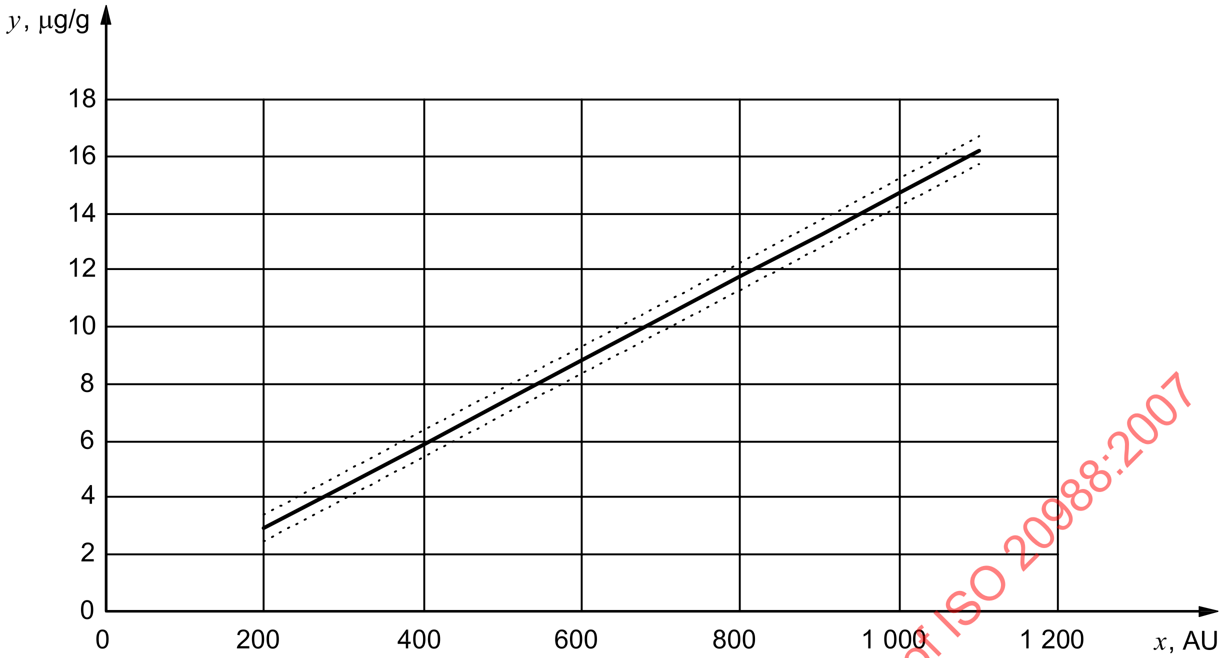


Key

x response, in area units (AU)

y_R benzene standard fraction, in micrograms per gram (µg/g)

Figure C.1 — Measured values (◇) of the calibration experiment and calibration line of gas chromatograph for benzene



Key

x response, in area units (AU)

y benzene fraction, in micrograms per gram (µg/g)

Figure C.2 — Analytical function and 95 % margins of uncertainty of gas chromatograph for benzene

C.5 Evaluation of toluene measuring method for use in industrial hygiene

This example demonstrates uncertainty estimation by analysis of a set of input data collected in evaluation of a measuring method for toluene in workplace atmospheres. For this purpose, M = 20 diffusive samplers of the same type were exposed to K = 5 different test-gas atmospheres of toluene. The aim of this test is to predict the uncertainty of results of measurement obtained in future application of a single diffusive sampler of the evaluated type in workplace atmospheres. The input data were taken from ISO 16107 [12]. This example demonstrates application of method A4 described in Annex B.5.

The work steps and the obtained results are summarized in Table C.8. The evaluated set of input data are given in Table C.9.

The analysis provided following results. The standard uncertainty of corrected toluene results y obtained by application of a diffusive sampler of the considered type in the range of 70 mg/m^3 $< y <$ 770 mg/m^3 is estimated by $w(y) = u(y)/y$ = 5,4 %. The expanded 95 % uncertainty $W_{0,95}(y)$ of corrected toluene results y obtained by application of a diffusive sampler of the considered type in the range of 70 mg/m^3 $< y <$ 770 mg/m^3 is estimated by $W_{0,95}(y)$ = 11 %.

Figure C.3 shows the calibration line for toluene. Figure C.4 shows the analytical function and the 95 % margins of uncertainty of this function. The reference values $y_R(k)$ used to establish the analytical function are encompassed completely by the 95 % margin of uncertainty $[y - U_{0,95}(y); y + U_{0,95}(y)]$.

The obtained uncertainty parameters are applicable to individual results of measurement y in the range of 70 mg/m^3 $< y <$ 770 mg/m^3 of toluene concentration in workplace atmospheres that can be obtained in future application of the evaluated method of measurement using a single sampler.

In addition, the following results were obtained. A 95 % confidence limit on the standard uncertainty $w(y)$ is estimated by $L((w(y))$ = 1,37 · $w(y)$ = 7,2 % using Equation (17) of 9.2.

A 95 % confidence limit on the expanded uncertainty $W_{0,95}(y)$ is given by $L(W_{0,95}(y))$ = 1,96 $L(w(y))$ = 14 %. In ISO 16107 [12], $L(W_{0,95}(y))$ is called "95 % confidence limit on the sampler accuracy".

The 95 % confidence limit on the expanded 95 % uncertainty $L(W_{0,95}(y))$ indicates that it is unlikely to find in another evaluation of the considered method of measurement of the same size (N = 20) an estimate of expanded 95 % uncertainty $W_{0,95}(y) > 14$ %.

Table C.8 — Work steps and results

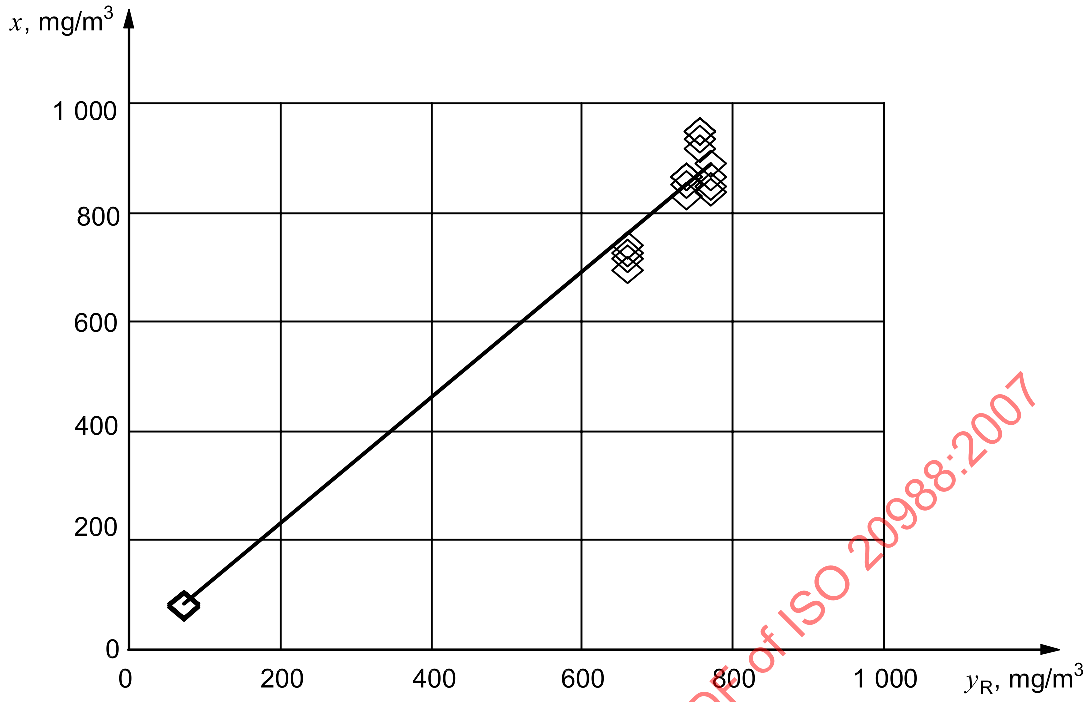
Step	Element	Instruction	Result
1	**Problem specification**		
	Method of measurement	Diffusive sampling by means of solid sorbent tube; desorption by means of a solvent, e.g. CS_2; analytical quantification by gas chromatography, FID [13], [14].	—
	Control conditions	Calibration of gas chromatograph and control of calibration state.	—
	Ambient conditions	As met in workplace atmospheres.	—
	Evaluated quantity	Result of measurement: 2 h average of toluene concentration in a workplace environment to which a worker can be exposed.	y
	Analytical function	$y = \frac{x}{b}$ where y is the result of measurement; x is the uncorrected output signal; b is the correction factor for bias in x.	—
	Uncertainty parameters	Relative standard uncertainty in the range of 70 mg/m^3 < y < 770 mg/m^3 .... Relative expanded uncertainty of results of measurement y on a level of coverage of 95 % in the range of 70 mg/m^3 < y < 770 mg/m^3	$w(y) = u(y)/y$ $W_{0,95}(y)$
	Experimental design	Type A4: N = 20 diffusive samplers of the same make were exposed in groups of M = 4 samplers in K = 5 different test-gas atmospheres.	—
	Input data	Series of observations $x(j)$ with j = 1 to N = 20 provided by parallel exposition of groups of M = 4 diffusive samplers in each of K = 5 test-gas atmospheres.	See Table C.9.
	Reference values	Series of reference values $y_R(j)$ with j = 1 to N = 20. According to the number M = 4 of samplers exposed to the same test gas, the same value is assigned four times to the series of reference values $y_R(j)$ with j = 1 to N = 20.	See Table C.9.
	Additional information	Relative standard uncertainty $u(y_R)/y_R$ .... Relative standard uncertainty $u(y)/y$ .... The uncertainty of the reference values is negligible.	$\leqslant$ 0,01 constant
	Representativeness	The experimental design used to collect the input data for uncertainty estimation resembles the intended future application in workplace atmosphere in good approximation.	—
	Effects not addressed	For the purpose of this example, additional sources of uncertainty were not considered.	—

Table C.8 (*continued*)

Step	Element	Instruction	Result
2	**Data treatment**		
	Model equation	$y = x / b$	—
	Variance equation	$\mathrm{var}(y) = \left(\frac{u(x)}{b}\right)^2 + \left(\frac{y \cdot u(b)}{b}\right)^2 - 2 \cdot \mathrm{cov}(x,b)$	—
	Covariance	$\mathrm{cov}(x,b)$	0
	Bias of y	Due to correction by factor b, ...	0 mg/m^3
	Model equation of evaluation	$x(j) = y_\mathrm{R}(j) \cdot (b + e_x(j))$ with relative deviation $e_x(j) = \frac{x(j)}{y_\mathrm{R}(j)} - b$	—
	Correction factor b	$b = \frac{1}{N} \sum_{j=1}^{N} \frac{x(j)}{y_\mathrm{R}(j)}$	1,14
	Residual standard deviation	$u(e_x) = s\left(\frac{x}{y_\mathrm{R}}\right) = \sqrt{\frac{1}{N-1} \sum_{j=1}^{N} \left(\frac{x(j)}{y_\mathrm{R}(j)} - b\right)^2}$	0,060
	Standard uncertainty of quantity x	$\frac{u(x)}{x} = \frac{u(e_x)}{b}$	0,054
	Standard uncertainty of b	$u(b) = \frac{u(e_x)}{\sqrt{N}}$	0,013
3	**Results of uncertainty analysis**		
	Standard uncertainty of y (relative)	$w(y) = \frac{u(y)}{y} = \frac{s\left(\frac{x}{y_\mathrm{R}}\right)}{b} \cdot \sqrt{1 + \frac{1}{N}}$	0,052
	Number of degrees of freedom	$\nu = N - 1$ .... since $\frac{u(y_\mathrm{R})}{y_\mathrm{R}} \approx s\left(\frac{x}{y_\mathrm{R}}\right) / b$	19
	Coverage factor	$k_{0,95}$	2,1
	Expanded 95 % uncertainty of y	$W_{0,95}(y) = k_{0,95} \cdot u(y) / y$	0,11
	95 % confidence limit on $w(y)$	$L(w(y)) = w(y)\sqrt{\nu / \chi^2(\gamma, \nu_\mathrm{eff})} = 1{,}37 \cdot w(y)$ .... $\gamma = 0{,}95$	0,072
	95 % confidence limit on $W_{0,95}(y)$	$L(W_{0,95}(y)) = 1{,}96 \cdot u(y) / y$ .... (95 % confidence limit on the sampler accuracy as described by ISO 16107)	0,14
	Range of application	$\min(y) \leqslant y \leqslant \max(y)$	70 mg/m^3 $\leqslant y \leqslant$ 770 mg/m^3

Table C.9 — Input data and results of measurement

Index j	Index test-gas k	Test-gas concentration y_R mg/m^3	Uncorrected response x mg/m^3	Result of measurement $y = x/b$ mg/m^3
1	1	73,14	84,99	74,3
2	1	73,14	80,67	70,5
3	1	73,14	77,67	67,9
4	1	73,14	83,96	73,4
5	2	658,6	725,8	634,6
6	2	658,6	716,6	626,5
7	2	658,6	738,3	645,5
8	2	658,6	695,5	608,1
9	3	738,7	829,6	725,3
10	3	738,7	865,0	756,3
11	3	738,7	865,0	756,3
12	3	738,7	850,2	743,3
13	4	755,9	948,7	829,4
14	4	755,9	935,0	817,5
15	4	755,9	947,9	828,7
16	4	755,9	917,7	802,3
17	5	771,1	862,9	754,4
18	5	771,1	890,6	778,6
19	5	771,1	847,4	740,9
20	5	771,1	836,6	731,4

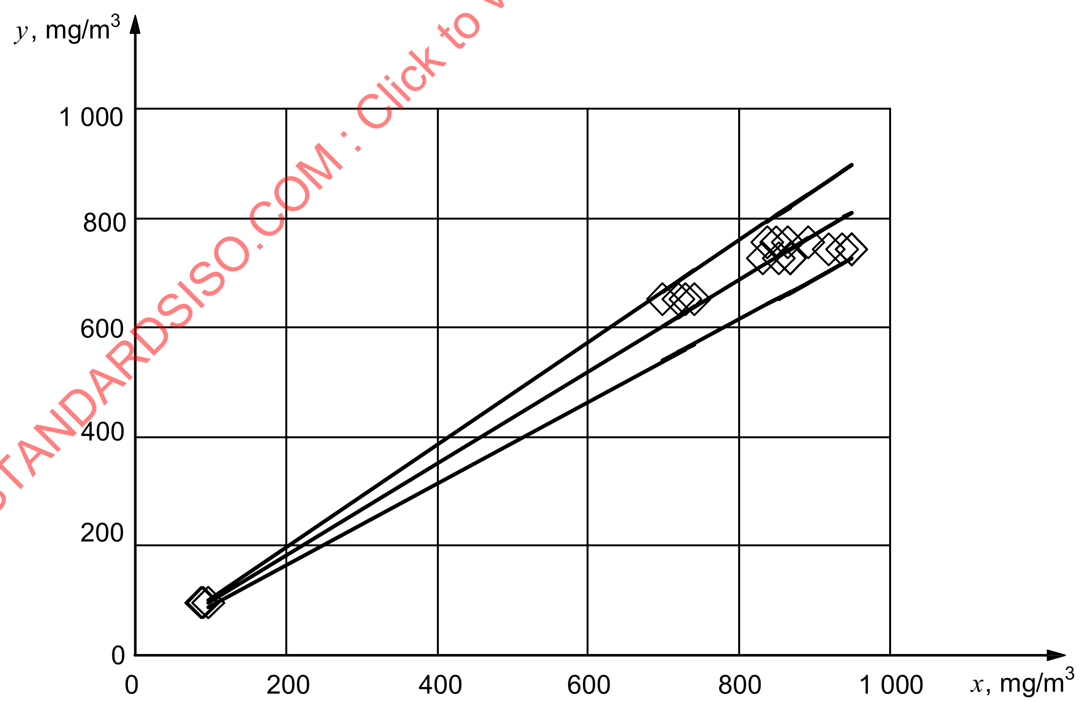


Key

x uncorrected response, in milligrams per cubic metre (mg/m^3)

y_R toluene test-gas concentration, in milligrams per cubic metre (mg/m^3)

Figure C.3 — Measured values (◇) of the calibration experiment and calibration line for toluene



Key

x uncorrected response, in milligrams per cubic metre (mg/m^3)

y toluene concentration, in milligrams per cubic metre (mg/m^3)

Figure C.4 — Measured values (◇) of the calibration experiment, analytical equation and 95 % margins of uncertainty

C.6 Calibration of an automated emission measuring system

This example demonstrates uncertainty estimation by evaluation of a set of input data provided in a regular calibration procedure of an automated emission measuring system (AMS) of dust in source emissions of a municipal waste incinerator at operating conditions (temperature range: 142 °C to 146 °C; moisture range: 14,3 % to 16,9 %; oxygen range: 11,1 % to 12,9 %). Reference values were provided by a reference method operated in parallel with the AMS at the same stack. Statistical evaluation was realized by method A5, case 1, described in Annex B.6.

The work steps and obtained results are summarized in Table C.10. The evaluated input data are given in Table C.11.

The analysis provided the following results. The standard uncertainty $u(y)$ of dust concentrations y obtained by the calibrated AMS at operating conditions within the range 1,2 mg/m^3 $< y <$ 8,5 mg/m^3 is found between 0,44 mg/m^3 and 0,53 mg/m^3. The expanded uncertainty $U_{0,95}(y)$ of dust concentrations y obtained by the calibrated AMS at operating conditions within the range 1,2 mg/m^3 $< y <$ 8,5 mg/m^3 is found between 0,9 mg/m^3 and 1,1 mg/m^3.

Figure C.5 provides a graphical impression of the calibration line and the corresponding 95 % margin of uncertainty. Due to the applied method of inverse regression, the analytical function coincides with the calibration line. The reference values y_R used to establish the calibration line are encompassed completely by the 95 % margin of uncertainty $[y - U_{0,95}(y); y + U_{0,95}(y)]$.

The uncertainty parameters are applicable to individual results of measurement y in the range of 1,2 mg/m^3 to 8,5 mg/m^3 obtained by the calibrated AMS at operating conditions identical to those during calibration (temperature range: 142 °C to 146 °C; moisture range: 14,3 % to 16,9 %; oxygen range: 11,1 % to 12,9 %). If results of measurement are reported for standard conditions (e.g. 0 °C, dry gas, 11,0 % O_2), the uncertainty parameters have to be converted to standard conditions. Then, the additional uncertainty contributions of the conversion parameters like temperature, moisture or oxygen content have to be taken into account additionally, e.g. by an indirect approach.