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Determination and use of straight-line calibration functions

Détermination et utilisation des fonctions d'étalonnage linéaire

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Foreword

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

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

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

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order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn.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again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TS 28037:2010 was prepared by Technical Committee ISO/TC 69, *Applications of statistical methods*, Subcommittee SC 6, *Measurement methods and results*.

Introduction

Calibration is an essential part of many measurement procedures and often involves fitting to measured data a calibration function that best describes the relationship of one variable to another. This Technical Specification considers straight-line calibration functions that describe a dependent variable Y as a function of an independent variable X. The straight-line relationship depends on the intercept A and the slope B of the line. A and B are referred to as the parameters of the line. The purpose of a calibration procedure is to determine estimates a and b of A and B for a particular measuring system under consideration on the basis of measurement data $(x_i,\ y_i)$, $i = 1, \ldots, m$, provided by the measuring system. The measurement data have associated uncertainty, which means there will be uncertainty associated with a and b. This Technical Specification describes how a and b can be determined given the data and the associated uncertainty information. It also provides a means for evaluating the uncertainties associated with these estimates. The treatment of uncertainty in this Technical Specification is carried out in a manner consistent with ISO/IEC Guide 98-3:2008, "Guide to the expression of uncertainty in measurement" (GUM).

Given the uncertainty information associated with the measurement data, an appropriate method can be specified to determine estimates of the calibration function parameters. This uncertainty information may include quantified covariance effects, relating to dependencies among some or all of the quantities involved.

Once the straight-line model has been fitted to the data, it is necessary to determine whether or not the model and data are consistent with each other. In cases of consistency, the model so obtained can validly be used to predict a value x of the variable X corresponding to a measured value y of the variable Y provided by the same measuring system. It can also be used to evaluate the uncertainties associated with the calibration function parameters and the uncertainty associated with the predicted value x.

The determination and use of a straight-line calibration function can therefore be considered to consist of five steps:

1 Obtaining uncertainty and covariance information associated with the measurement data – although dependent on the particular area of measurement, examples are provided within this Technical Specification;

2 Providing best estimates of the straight-line parameters;

3 Validating the model, both in terms of the functional form (does the data reflect a straight-line relationship?) and statistically (is the spread of the data consistent with their associated uncertainties?) using a chi-squared test;

4 Obtaining the standard uncertainties and covariance associated with the estimate of the straight-line parameters.

5 Using the calibration function for prediction, that is, determining an estimate x of the X-variable and its associated uncertainty corresponding to a measured value y of the Y-variable and its associated uncertainty.

The above steps are shown diagrammatically in Figure 1.

The main aim of this Technical Specification is to consider steps 2 to 5. Therefore, as part of step 1, before using this Technical Specification, the user will need to provide standard uncertainties, and covariances if relevant, associated with the measured Y-values and, as appropriate, those associated with the measured X-values. Account should be taken of the principles of the GUM in evaluating these uncertainties on the basis of a measurement model that is specific to the area of concern.

ISO 11095:1996 [14] is concerned with linear calibration using reference materials. It differs from this Technical Specification in the ways given in Table 1.

The numerical methods given are based on reference [6].

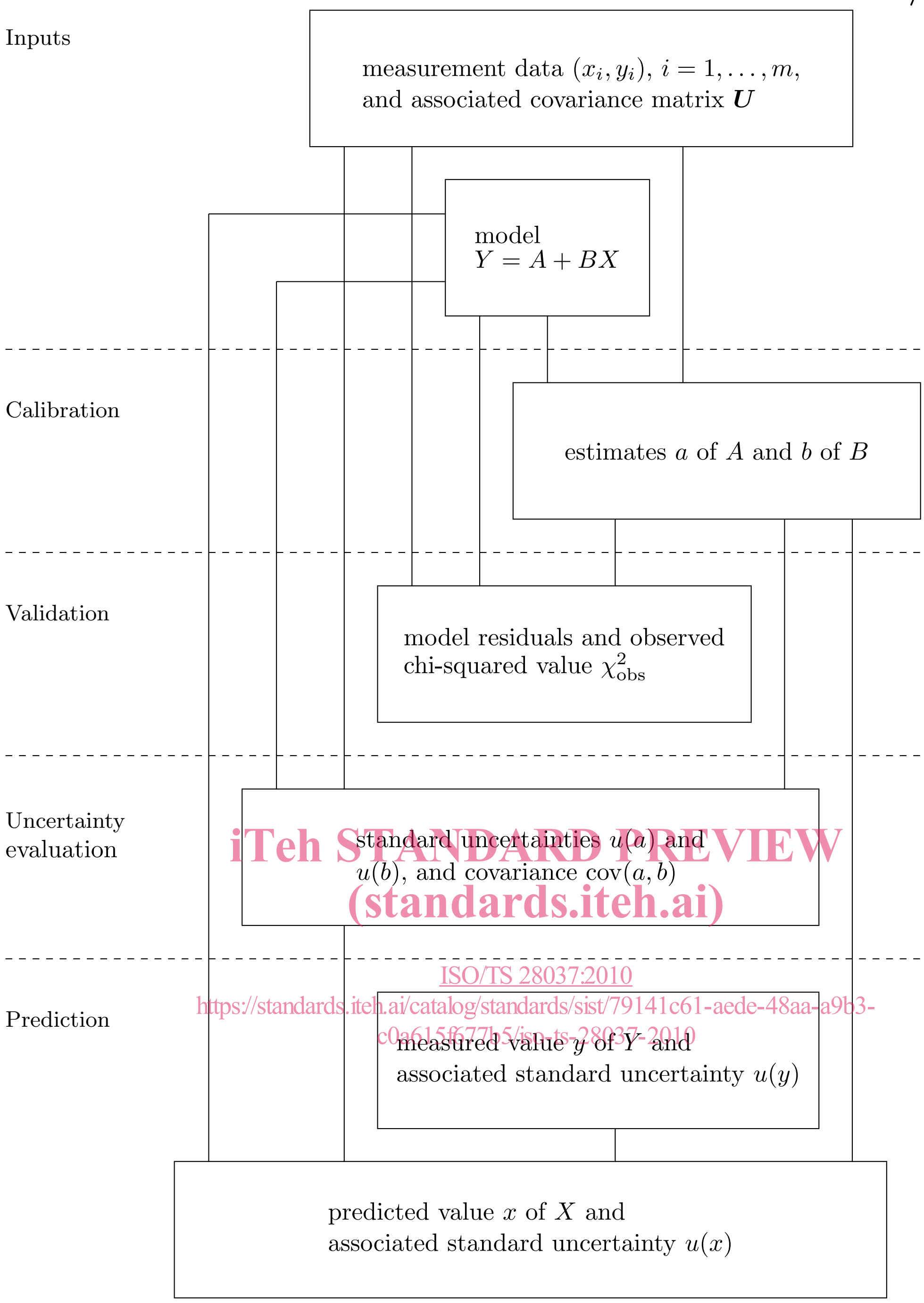

Figure 1 — Summary of the steps in the determination and use of straight-line calibration functions

Table 1 — Differences between ISO 11095:1996 and ISO/TS 28037:2010

Feature	ISO 11095:1996	ISO/TS 28037:2010
Specifically addresses reference materials	Yes	More general
X-values assumed to be known exactly	Yes	More general uncertainty information
All measured values obtained independently	Yes	More general uncertainty information
Terminology aligned with GUM	No	Yes
Types of uncertainty structure treated	Two	Five, including the most general case
Only uncertainty associated with random errors	Yes	More general uncertainty information
Consistency test	ANOVA	Chi-squared
Uncertainty associated with predictions	Ad hoc	GUM compatible

Determination and use of straight-line calibration functions

1 Scope

This Technical Specification is concerned with linear, that is, straight-line, calibration functions that describe the relationship between two variables X and Y, namely, functions of the form $Y = A + BX$. Although many of the principles apply to more general types of calibration function, the approaches described exploit the simple form of the straight-line calibration function wherever possible.

Values of the parameters A and B, are determined on the basis of measured data points $(x_i,\ y_i)$, $i = 1, \ldots, m$. Various cases are considered relating to the nature of the uncertainties associated with these data. No assumption is made that the errors relating to the y_i are homoscedastic (having equal variance), and similarly for the x_i when the errors are not negligible.

Estimates of the parameters A and B are determined using least squares methods. The emphasis of this Technical Specification is on choosing the least squares method most appropriate for the type of measurement data, in particular methods that reflect the associated uncertainties. The most general type of covariance matrix associated with the measurement data is treated, but important special cases that lead to simpler calculations are described in detail.

For all cases considered, methods for validating the use of the straight-line calibration functions and for evaluating the uncertainties and covariance associated with the parameter estimates are given.

The Technical Specification also describes the use of the calibration function parameter estimates and their associated uncertainties and covariance to predict a value of X and its associated standard uncertainty given a measured value of Y and its associated standard uncertainty.

NOTE 1 The Technical Specification does not give a general treatment of outliers in measurement data, although the validation tests given can be used as a basis for identifying discrepant data.

NOTE 2 The Technical Specification describes a method to evaluate the uncertainties associated with the measurement data in the case that those uncertainties are known only up to a scale factor (Annex E).

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 99:2007, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/IEC Guide 98-3:2008 and ISO/IEC Guide 99:2007 and the following apply.

A glossary of principal symbols is given in Annex G.

3.1
measured quantity value
quantity value representing a measurement result

[ISO/IEC Guide 99:2007 2.10]

3.2
measurement uncertainty
non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

[ISO/IEC Guide 99:2007 2.26]

3.3
standard measurement uncertainty
measurement uncertainty expressed as a standard deviation

[ISO/IEC Guide 99:2007 2.30]

3.4
covariance associated with two quantity values
parameter characterizing the interdependence of the quantity values being attributed to two measurands, based on the information used

3.5
measurement covariance matrix
covariance matrix
matrix of dimension $N \times N$ associated with a vector estimate of a vector quantity of dimension $N \times 1$, containing on its diagonal the squares of the standard uncertainties associated with the respective components of the vector estimate of the vector quantity, and, in its off-diagonal positions, the covariances associated with pairs of components of the vector estimate of the vector quantity

NOTE 1 A covariance matrix $\boldsymbol{U_x}$ of dimension $N \times N$ associated with the vector estimate $\boldsymbol{x}$ of a vector quantity $\boldsymbol{X}$ has the representation

$$\boldsymbol{U_x} = \begin{bmatrix} \mathrm{cov}(x_1, x_1) & \cdots & \mathrm{cov}(x_1, x_N) \\ \vdots & \ddots & \vdots \\ \mathrm{cov}(x_N, x_1) & \cdots & \mathrm{cov}(x_N, x_N) \end{bmatrix},$$

where $\mathrm{cov}(x_i, x_i) = u^2(x_i)$ is the variance (squared standard uncertainty) associated with x_i and $\mathrm{cov}(x_i, x_j)$ is the covariance associated with x_i and x_j. $\mathrm{cov}(x_i, x_j) = 0$ if elements X_i and X_j of $\boldsymbol{X}$ are uncorrelated.

NOTE 2 Covariances are also known as mutual uncertainties.

NOTE 3 A covariance matrix is also known as a variance-covariance matrix.

NOTE 4 Definition adapted from ISO/IEC Guide 98-3:2008/Suppl. 1:2008, definition 3.11 [13].

3.6
measurement model
mathematical relation among all quantities known to be involved in a measurement

[ISO/IEC Guide 99:2007 2.48]

3.7
functional model
statistical model involving errors associated with the dependent variable

3.8
structural model
statistical model involving errors associated with the independent and dependent variables

3.9
calibration
operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication

NOTE 1 A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases, it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

NOTE 2 Calibration should not be confused with adjustment of a measuring system, often mistakenly called 'self-calibration', nor with verification of calibration.

NOTE 3 Often the first step alone in the above definition is perceived as being calibration.

[ISO/IEC Guide 99:2007 2.39]

3.10
probability distribution
⟨random variable⟩ function giving the probability that a random variable takes any given value or belongs to a given set of values

NOTE 1 The probability on the whole set of values of the random variable equals 1.

NOTE 2 A probability distribution is termed univariate when it relates to a single (scalar) random variable, and multivariate when it relates to a vector of random variables. A multivariate probability distribution is also described as a joint distribution.

NOTE 3 A probability distribution can take the form of a distribution function or a probability density function.

NOTE 4 Definition and note 1 adapted from ISO 3534-1:1993, definition 1.3 and ISO/IEC Guide 98-3:2008, definition C.2.3; notes 2 and 3 adapted from ISO/IEC Guide 98-3:2008/Suppl. 1:2008, definition 3.1 [13].

3.11
normal distribution
probability distribution of a continuous random variable X having the probability density function

$$g_X(\xi) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{1}{2}\left(\frac{\xi-\mu}{\sigma}\right)^2\right],$$

for $-\infty < \xi < +\infty$

NOTE 1 μ is the expectation and σ is the standard deviation of X.

NOTE 2 The normal distribution is also known as the Gaussian distribution.

NOTE 3 Definition and note 1 adapted from ISO 3534-1:1993, definition 1.37; note 2 adapted from ISO/IEC Guide 98-3:2008, definition C.2.14.

3.12
***t*-distribution**
probability distribution of a continuous random variable X having the probability density function

$$g_X(\xi) = \frac{\Gamma\left(\dfrac{\nu+1}{2}\right)}{\sqrt{\pi\nu}\,\Gamma(\nu/2)}\left(1+\frac{\xi^2}{\nu}\right)^{-(\nu+1)/2},$$

for $-\infty < \xi < +\infty$, with parameter ν, a positive integer, the degrees of freedom of the distribution, where

$$\Gamma(z) = \int_0^\infty t^{z-1} e^{-t}\,\mathrm{d}t, \qquad z > 0,$$

is the gamma function

[ISO/IEC Guide 98-3:2008/Suppl. 1:2008 3.5]

3.13
chi-squared distribution
χ^2 distribution
probability distribution of a continuous random variable X having the probability density function

$$g_X(\xi) = \frac{\xi^{(\nu/2)-1}}{2^{\nu/2}\Gamma(\nu/2)} \exp\left(-\frac{\xi}{2}\right),$$

for $0 \le \xi < \infty$, with parameter ν, a positive integer, where Γ is the gamma function

NOTE The sum of the squares of ν independent standardized normal variables is a χ^2 random variable with parameter ν; ν is then called the degrees of freedom.

3.14
positive definite matrix

matrix $\boldsymbol{M}$ of dimension $n \times n$ having the property $\boldsymbol{z}^\top \boldsymbol{M}\boldsymbol{z} > 0$ for all non-zero vectors $\boldsymbol{z}$ of dimension $n \times 1$

3.15
positive semi-definite matrix

matrix $\boldsymbol{M}$ of dimension $n \times n$ having the property $\boldsymbol{z}^\top \boldsymbol{M}\boldsymbol{z} \ge 0$ for all non-zero vectors $\boldsymbol{z}$ of dimension $n \times 1$

4 Conventions and notation

For the purpose of this Technical Specification the following conventions and notations are adopted.

4.1 X is termed the independent variable and Y the dependent variable even when the knowledge of X and Y is 'interchangeable', as in Clause 7, for example.

4.2 The quantities A and B are termed the parameters of the straight-line calibration function $Y = A + BX$. A and B are also used to denote (dummy) variables in expressions involving the calibration function parameters.

4.3 The quantities X_i and Y_i are used as (dummy) variables to denote the co-ordinates of the ith data point.

4.4 The constants A^* and B^* are (unknown) values of A and B that specify the straight-line calibration function $Y = A^* + B^*X$ for a particular measuring system under consideration.

4.5 The constants X_i^* and Y_i^* are the (unknown) co-ordinates of the ith data point provided by the measuring system satisfying $Y_i^* = A^* + B^*X_i^*$.

4.6 x_i and y_i are the measured values of the co-ordinates of the ith data point.

4.7 a and b are estimates of the calibration function parameters for the measuring system.

4.8 x_i^* and y_i^* are estimates of the co-ordinates of the ith data point satisying $y_i^* = a + bx_i^*$.

4.9 A vector of dimension $m \times 1$ is denoted thus:

$$\boldsymbol{x} = \begin{bmatrix} x_1 \\ \vdots \\ x_m \end{bmatrix}, \qquad \boldsymbol{x}^\top = \begin{bmatrix} x_1 & \dots & x_m \end{bmatrix},$$

and a matrix of dimension $m \times n$ is denoted thus:

$$\boldsymbol{A} = \begin{bmatrix} a_{11} & \dots & a_{1n} \\ \vdots & \ddots & \vdots \\ a_{m1} & \dots & a_{mn} \end{bmatrix}, \qquad \boldsymbol{A}^\top = \begin{bmatrix} a_{11} & \dots & a_{m1} \\ \vdots & \ddots & \vdots \\ a_{1n} & \dots & a_{mn} \end{bmatrix}.$$

The dimension of the vector or matrix is always specified to avoid possible confusion.

4.10 $\top$ denotes transpose.

4.11 The zero matrix is denoted by **0** and the unit vector is denoted by **1**.

4.12 Some symbols have more than one meaning. The context clarifies the usage.

4.13 Numbers displayed in tables to a fixed number of decimal places are correctly rounded representations of numbers stored to higher precision, as would be the case in a spreadsheet, for example. Therefore, minor inconsistencies may be perceived between displayed column sums and the column sums of the displayed numbers.

4.14 In some tables, a subclause number above a column or columns indicates where the formula is given for determining the values below.

4.15 In the examples, while data values are provided to a given precision, the results of calculations are provided to a higher precision to allow the user to compare results when undertaking the calculations.

5 Principles of straight-line calibration

5.1 General

5.1.1 This clause considers how a relationship $Y = A + BX$ describing the dependent variable Y (also called 'response') as a function of the independent variable X (also called 'stimulus') can be determined from measurement data. In the context of calibration, the measurement data arise when a measuring instrument specified by (unknown) values A^* and B^* of the calibration function parameters is 'stimulated' by artefacts with calibrated values of X_i given in standard units, of a property of the artefacts, and the corresponding 'responses' or indications Y_i of the instrument are recorded. The relationship provides the response Y of the system given an artefact with calibrated quantity X. This process is termed 'forward evaluation'. More useful in practice, the relationship allows a measured response y of Y to be converted to an estimate x, in standard units, of the property X of an artefact. This process is termed 'inverse evaluation' or 'prediction'.

5.1.2 The calibration of a measuring system should take into account measurement uncertainties, and, if present, covariances associated with the measurement data. The output of a calibration procedure is a calibration function to be used for prediction (and, if required, forward evaluation). The output also includes the standard uncertainties and covariance associated with the estimates a and b of the parameters describing the calibration function, which are used to evaluate the standard uncertainties associated with prediction (and forward evaluation).

5.2 Inputs to determining the calibration function

5.2.1 Measurement data

The information required to determine the straight-line calibration function are the measurement data and their associated standard uncertainties and covariances. In this Technical Specification, the measurement data are denoted by (x_i, y_i), $i = 1, \ldots, m$, that is, m pairs of measured values of X and Y. It is assumed that m is at least two and that the values of x_i are not all equal to each other.

NOTE The uncertainties associated with the estimates a and b generally decrease as m increases. Therefore, calibration should aim to use as many measured data points as is economically viable.

5.2.2 Associated uncertainties and covariances

The standard uncertainties associated with x_i and y_i are denoted by $u(x_i)$ and $u(y_i)$ respectively. The covariance associated with x_i and x_j is denoted by $\text{cov}(x_i, x_j)$. Similarly, those associated with y_i and y_j, and with x_i and y_j, are denoted by $\text{cov}(y_i, y_j)$ and $\text{cov}(x_i, y_j)$, respectively. Annex D indicates how the uncertainties and covariances associated with the measured response and stimulus variables can be evaluated and gives an interpretation of that uncertainty information. The complete uncertainty information is represented by an array of elements (matrix) $\boldsymbol{U}$ of dimension $2m \times 2m$ holding the variances (squared standard uncertainties) $u^2(x_i)$ and $u^2(y_i)$ and the covariances:

$$\boldsymbol{U} = \begin{bmatrix} u^2(x_1) & \ldots & \text{cov}(x_1, x_m) & \text{cov}(x_1, y_1) & \ldots & \text{cov}(x_1, y_m) \\ \vdots & \ddots & \vdots & \vdots & \ddots & \vdots \\ \text{cov}(x_m, x_1) & \ldots & u^2(x_m) & \text{cov}(x_m, y_1) & \ldots & \text{cov}(x_m, y_m) \\ \text{cov}(y_1, x_1) & \ldots & \text{cov}(y_1, x_m) & u^2(y_1) & \ldots & \text{cov}(y_1, y_m) \\ \vdots & \ddots & \vdots & \vdots & \ddots & \vdots \\ \text{cov}(y_m, x_1) & \ldots & \text{cov}(y_m, x_m) & \text{cov}(y_m, y_1) & \ldots & u^2(y_m) \end{bmatrix}.$$

For many applications, some or all covariances are taken as zero (see 5.3).

NOTE This Technical Specification is concerned with problems in which the $u(x_i)$ or the $u(y_i)$ are generally different.

5.3 Determining the calibration function

5.3.1 The inputs to determining the calibration function are the measurement data and their associated uncertainties and possibly covariances. Given parameters A and B, the inputs can be used to provide a measure of the departure of the ith data point (x_i, y_i) from the line $Y = A + BX$. The estimates a and b are determined by minimizing a sum of squares of these departures, or a more general measure when any covariances are non-zero. How this is achieved depends on the 'uncertainty structure' associated with the measurement data. This uncertainty structure relates to the answers to the following questions:

i) Are the uncertainties associated with the measured values x_i negligible?

ii) Are the covariances associated with pairs of measured values negligible?

5.3.2 The following cases, given in increasing order of complexity and depending on the answers to the questions in 5.3.1, are considered in this Technical Specification:

a) The only uncertainties are associated with the measured values y_i and all covariances associated with the data are regarded as negligible (Clause 6);

b) Uncertainties are associated with the measured values x_i and y_i and all covariances associated with the data are regarded as negligible (Clause 7);

c) Uncertainties are associated with the measured values x_i and y_i and the only covariances are associated with the pairs (x_i, y_i) (Clause 8);

d) The only uncertainties are associated with the measured values y_i and the only covariances are associated with the y_i and the y_j $(i \neq j)$ (Clause 9);

e) The most general case in which there are uncertainties associated with the measured values x_i and y_i and covariances associated with all pairs of values of the x_i, the x_j, the y_k and the y_ℓ (Clause 10).

5.3.3 For each case in 5.3.2 are given

a) the prescribed measurement data and uncertainty structure,

b) the corresponding statistical model,

c) the least squares problem addressed,

d) the calculation steps,

e) properties of the statistical model,

f) validation of the model,

g) organization of the calculations for the computer, where appropriate,

h) a numerical algorithm, where appropriate, and

i) one or more worked examples.

5.4 Numerical treatment

In Annex C, a general approach to the most general case e) in 5.3.2 is given. It can be used to treat all the other cases and uses sophisticated, numerically stable methods. The cases a) to c) in 5.3.2 can, however, be treated using elementary operations, which can be implemented in a spreadsheet, for example. The cases d) and e) in 5.3.2 require some matrix operations, which are straightforward to implement in a computer language supporting matrix arithmetic, but are not well suited to spreadsheet calculations.

5.5 Uncertainties and covariance associated with the calibration function parameters

5.5.1 For all cases considered, estimates of the calibration function parameters can be expressed (explicitly or implicitly) as functions of the measurement data. The principles of the GUM [ISO/IEC Guide 98-3:2008] can be applied to propagate the uncertainties and covariances associated with the measurement data through these functions to obtain those associated with these parameter estimates. In this way, the measurement data are used to provide estimates a and b of the calibration function parameters, and to evaluate standard uncertainties $u(a)$ and $u(b)$ and the covariance $\mathrm{cov}(a, b)$ associated with these estimates. For the cases a) and d) in 5.3.2, the propagation is exact since the parameter estimates can be expressed as linear combinations of the inputs y_i. For the other cases, in which the parameter estimates cannot be so expressed, the propagation is approximate, based on a linearization about the parameter estimates. For many purposes, the approximation incurred by the linearization will be sufficiently accurate.

NOTE When the propagation of uncertainty is approximate, and particularly if the uncertainties involved are large (for example, in some areas of biological measurement), an approach based on the propagation of distributions can be employed. This approach [ISO/IEC Guide 98-3:2008/Suppl. 1:2008] uses a Monte Carlo method (not treated in this Technical Specification).

5.5.2 The primary outputs in describing the straight-line calibration function are the parameter estimate vector $\boldsymbol{a}$ of dimension 2×1 and the covariance matrix $\boldsymbol{U_a}$ of dimension 2×2 given by

$$\boldsymbol{a} = \begin{bmatrix} a \\ b \end{bmatrix}, \qquad \boldsymbol{U_a} = \begin{bmatrix} u^2(a) & \mathrm{cov}(a, b) \\ \mathrm{cov}(b, a) & u^2(b) \end{bmatrix}, \tag{1}$$

where $u(a)$ and $u(b)$ are the standard uncertainties associated with a and b, respectively, and $\mathrm{cov}(a, b) = \mathrm{cov}(b, a)$ is the covariance associated with a and b.