



**SIM Guidelines on
the calibration of
non-automatic
weighing
instruments with
resolution less than
0.010 mg**

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SIM Guidelines on the calibration of non-automatic weighing instruments with resolution less than 0.010 mg

INDEX

1	BACKGROUND	5
2	AUTHORS	6
3	ACKNOWLEDGMENTS	7
4	INTRODUCTION	8
5	SCOPE	8
6	TERMINOLOGY AND SYMBOLS	9
7	GENERAL ASPECTS OF CALIBRATION	10
7.1	ELEMENTS OF CALIBRATION	10
7.1.1	<i>Range of calibration</i>	10
7.1.2	<i>Place of calibration</i>	10
7.1.3	<i>Preconditions, preparations</i>	11
7.2	REFERENCE AND AUXILIARY WEIGHTS	12
7.3	AUXILIARY EQUIPMENT	13
8	MEASUREMENT METHOD	14
8.1	INDICATION	14
8.2	DESCRIPTION OF THE METHOD	15
8.2.1	<i>Operation routine</i>	16
9	MEASUREMENT RESULTS	17
9.1	MEASUREMENT MODEL	17
10	UNCERTAINTY CALCULATION OF THE ESTIMATES OF ERRORS OF INDICATION AND CONVENTIONAL MASS CORRECTIONS OF AUXILIARY WEIGHTS	20
10.1	MATRIX U_{rep}	20
10.2	MATRIX U_{res}	21
10.3	MATRIX U_{resid}	22
10.4	MATRIX U_b	23
10.5	MATRIX U_{mR}	24
11	TRACEABILITY OF MEASUREMENTS	25
11.1	GOOD MEASUREMENT PRACTICES	25
12	REFERENCES	26
13	APPENDICES	27
13.1	APPENDIX A	27
13.1.1	<i>Appendix A1: EXAMPLE</i>	27
13.1.1.1	Initial information	27
13.1.1.2	Measurements	28
13.1.1.3	Calculation of estimates	32
13.1.1.4	Uncertainty calculation	42
13.1.2	<i>Appendix A 2: MATRIX MODELS</i>	54
13.2	APPENDIX B: SUGGESTIONS FOR AIR DENSITY ESTIMATION	56
13.2.1	<i>Formulas for air density</i>	56
13.2.1.1	Simplified version of the CIPM formula, exponential version	56
13.2.1.2	Average air density	56
13.2.1.3	Variations of the parameters of the air density components	57
13.2.2	<i>Air density uncertainty</i>	59
13.3	APPENDIX C: COVERAGE FACTOR k FOR EXPANDED MEASUREMENT UNCERTAINTY	60
13.3.1	<i>Objective</i>	60

13.3.2	<i>Normal distribution and sufficient reliability</i>	60
13.3.3	<i>Normal distribution, no sufficient reliability</i>	60
13.3.4	<i>Determining k for non-normal distributions considered as normal</i>	61
13.4	APPENDIX D: TERMS	62

1 BACKGROUND

This guide was developed as part of the activities of the project "*Calibration of weighing instruments - Microbalances*" within the macro project "*Strengthening National Metrology Institutes in the Hemisphere, in support of emerging technologies*" funded by the Inter-American Development Bank (IDB) for the National Metrology Institutes (NMIs) of the Inter-American Metrology System (SIM) and the Designated Institutes (DIs) for the development of technical research capabilities in metrology related to emerging technologies such as advanced manufacturing, nanotechnology and biotechnology.

One of the purposes of this project was the validation of an alternative method for the calibration of non-automatic weighing instruments with resolution less than 0.010 mg (microbalances or ultra-microbalances), instruments that have been incorporated into the field of emerging technologies in their processes. This alternative method provides calibration uncertainties in the order of micrograms or tenths of micrograms, which are lower than those that can be obtained by the direct comparison method with reference weights.

This guide is the result of the validation of the alternative method and contains the harmonized criteria so that it can be used as a reference document for calibration laboratories and accreditation bodies.

The NMIs participating in the project are (in alphabetical order):

- Centro de Estudios de Medición y Certificación de Calidad (CESMEC) – Chile
- Centro Nacional de Metrología (CENAM) – México
- Instituto Nacional de Calidad (INACAL) – Perú
- Instituto Nacional de Metrología (INM) – Colombia**
- Instituto Nacional de Tecnología Industrial (INTI) – Argentina
- Laboratorio Costarricense de Metrología (LCM) – Costa Rica**
- Laboratorio Tecnológico del Uruguay (LATU) – Uruguay

** These institutes received financing from sources other than the IDB.

2 **AUTHORS**

The people who participated in the development of this guide (in alphabetical order) are:

- | | |
|-----------------------------------|------------------|
| • Daniel González Cartagena | LATU - Uruguay |
| • Donny Taipe Araujo | INACAL - Peru |
| • Fernando Andrés García González | CESMEC - Chile |
| • Jorge Carlos Sanchez | INTI - Argentina |
| • Jhon J. Escobar Soto | INM - Colombia |
| • Juan Pablo García González | CESMEC - Chile |
| • Luis Omar Becerra Santiago | CENAM - Mexico |
| • Luis Manuel Peña | CENAM - Mexico |
| • Luz Marina Cori Almonte | INACAL - Peru |
| • Marcela Prendas Peña | LCM - Costa Rica |
| • Rubén Antonio Quille Ramos | INTI - Argentina |
| • Sandra Magaly Ramirez Jimenez | CENAM - Mexico |
| • Sheila Vanessa Preste Perez | LATU - Uruguay |

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4 INTRODUCTION

Non-automatic weighing instruments are widely used to determine the magnitude of a load in terms of its mass. While, for some applications specified by national legislations, instruments are subject to legal metrological control, e.g. type approval, inspection, verification, etc., there is an increasing need to have metrological quality certified by calibration, e.g. as required by ISO 9001 or ISO/IEC 17025.

5 SCOPE

This document contains guidance for the static calibration of non-automatic self-indicating weighing instruments with a resolution of less than 0.010 mg (hereinafter referred to as "instruments"), in particular for

1. measurements to be performed,
2. calculation of measurement results,
3. determination of measurement uncertainty,

The object of calibration is the indication provided by the instrument in response to an applied load. The results are expressed in units of mass. The value of the load indicated by the instrument is affected by the force due to gravity, the temperature and density of the load, and the temperature and density of the ambient air.

The measurement uncertainty depends significantly on the properties of the instrument to be calibrated itself, not solely on the calibration laboratory equipment; it can be reduced to some extent by increasing the number of measurements made for calibration. This guide does not specify upper or lower limits for measurement uncertainty.

It is up to the calibration laboratory and the customer to agree prior to calibration on the measurement uncertainty value appropriate for the use of the instrument, as well as the cost of calibration.

The objective of this Guide is to present an alternative calibration method applicable to instruments with a resolution of less than 0.010 mg, in addition to the recommendations provided by the SIM Guide for the calibration of non-automatic weighing instruments SIM MWG7/cg-01 [2], and to provide general

recommendations for the establishment of calibration procedures to minimize calibration uncertainties at small measurement intervals. The results of these can be considered equivalent within SIM member organizations.

Any such procedure should include a specified number of loads, determination of the indication errors and the measurement uncertainty associated with them. The test procedure should resemble as closely as possible the user's normal weighing operations

The information presented in this guide is intended for use by:

1. calibration laboratory accreditation bodies
2. accredited laboratories for the calibration of non-automatic weighing instruments with a resolution of less than 0.010 mg,
3. testing houses, laboratories or manufacturers using non-automatic weighing instruments calibrated to a resolution of less than 0.010 mg, used to perform measurements relevant to the quality of production that affect Quality System requirements (e.g. ISO 9000 series, ISO 10012, ISO/IEC 17025).

6 TERMINOLOGY AND SYMBOLS

The terminology used in this document is mainly based on the following documents:

- GUM [1] for terms related to the determination of results and measurement uncertainty,
- OIML R111 [5] for terms related to standard weights,
- OIML R76 [6] for terms related to the performance, construction and metrological characterization of non-automatic weighing instruments.
- VIM [7] for calibration-related terms

The terms that are not explained in this document, the references will be indicated in the place where they appear for the first time.

Symbols whose meaning is not self-explanatory shall be explained where they are used for the first time.

7 GENERAL ASPECTS OF CALIBRATION

7.1 Elements of calibration

Calibration consists of

1. the application of test loads to the instrument under specified conditions,
2. the determination of the error or variation of the indication, and
3. the estimation of the measurement uncertainty to be attributed to the results.

This method allows the calibration of an instrument with smaller uncertainties than those that can be achieved following the recommendations of the SIM MGW7/cg Guidelines [2]. However, its application requires a larger number of measurements and the implementation of a more complex mathematical technique, which makes the calibration process more extensive.

7.1.1 Range of calibration

The methodology proposed here is recommended for the calibration of a specific weighing interval, limited by a minimum load Min' and the largest load to be weighed Max' , which are expected to be provided by the customer.

The application of this methodology is not recommended for the full weighing range of the instrument, from zero to maximum capacity Max . Calibration results should not be extrapolated to values outside the agreed calibration range

Examples of matrix models and suggested loads for different weighing intervals can be found in Appendix A.

Appendix D contains definitions of the terms used in this section.

7.1.2 Place of calibration

Calibration is normally performed at the place where the instrument is used.

If an instrument is moved to another location after calibration, possible effects due to:

1. difference in local gravity acceleration,
2. variation in environmental conditions,
3. mechanical and thermal conditions during transport

may very likely alter the performance of the instrument and possibly invalidate the calibration.

7.1.3 Preconditions, preparations

Calibration should not be performed unless

1. the instrument can be clearly identified,
2. all instrument functions are free from the effects of contamination or damage and the essential functions necessary for calibration operate as intended,
3. the presentation of the weighing values is unambiguous and the indications, where given, are easily readable,
4. the normal conditions of use (air flow, vibrations, stability of the weighing location, etc.) are appropriate for the instrument to be calibrated,
5. the instrument is energized for a period before calibration, i.e., an appropriate time for the instrument to warm up, or as adopted by the customer,
6. the instrument is levelled,
7. the instrument has been exercised by placing a load close to the maximum load at least once, it is recommended to repeat several weighings.

Instruments should be adjusted before and during calibration. The adjustment should be performed with the means normally applied by the customer and following the manufacturer's instructions, when available.

As far as relevant to the calibration results, the status of the software settings, which could be altered by the customer, should be noted.

Instruments equipped with the automatic zeroing function or a zero indicating device [6] should be calibrated with the device operating or turned off, as determined by the customer.

For on site calibration the user of the instrument should be asked to ensure that normal conditions of use prevail during calibration. In this way disturbing effects such as air flows, vibrations or the inclination of the measuring platform can, as far as possible, be intrinsic to the measured values and can therefore be included in the determined measurement uncertainty.

Measurements should be made after an appropriate acclimatization period,

taking into account the recommendations in Appendix F of SIM Guidelines MWG7/cg-01 [2]. If the available space inside the instrument cabinet allows it, it is recommended to place the weights inside the cabinet.

Before placing the weights, the surface of the instrument plate should be carefully cleaned.

Measurements should be programmed in such a way that they are not interrupted until a series of measurements is completed. Depending on the performance of the instrument (stabilization time of the indications) a series of comparisons may take 2 to 3 hours.

7.2 Reference and auxiliary weights

For the selection of the standard weights for the application of this method, it is necessary to take into account the following:

1. A standard weight of mass equal or close to the maximum capacity of the weighing instrument must be available (*Max*).
2. If a different calibration range is chosen in agreement with the customer, the standard weight must have a mass value equal or close to the maximum point of the agreed calibration range (*Max'*).
3. The traceability of the weights to be used as standards should be achieved by calibration which consists in
 - the determination of the corresponding conventional mass value m_c or the correction δm_c to its nominal value m_N : $\delta m_c = m_c - m_N$, and the expanded uncertainty of the calibration U_{95} , or
 - confirmation that m_c is within the specified maximum permissible errors *emp*
$$emp: m_N - (emp - U_{95}) < m_c < m_N + (emp - U_{95})$$

In addition, the mass standards should meet the following requirements as much as appropriate considering their accuracy:

- the density ρ_s close enough to $\rho_c = 8\,000\text{ kg m}^{-3} = 8\text{ g cm}^{-3}$.
- appropriate surface finish to avoid mass change due to contamination by dirt or adhesion layers
- suitable magnetic properties so that interaction with the instrument to be calibrated is minimized.

Weights complying with the relevant specifications for accuracy class E_1 of the

international recommendation OIML R 111:2004 [5] should satisfy all these requirements.

4. It is necessary to use auxiliary weights of intermediate values of the calibration range of the instrument. Although these do not require calibration, it is recommended that they meet the physical characteristics of accuracy class E₂ weights or better.
5. The volume (or density) value and associated uncertainty of all weights involved in the calibration (auxiliary and reference weights) must be available

Examples of auxiliary weight selection can be found in Appendix A of this document.

7.3 Auxiliary equipment

The calibration laboratory must have the necessary auxiliary equipment for the calibration of the instrument.

Depending on the calibration uncertainty, the laboratory must have adequate measuring instruments to record the values of the environmental conditions (temperature, pressure and relative humidity when this is required depending on the required uncertainty in air density); these instruments must be calibrated.

8 MEASUREMENT METHOD

Tests are usually performed to determine

- the repeatability of the indications,
- the errors of indications,
- the effect of the eccentric application of a load on the indication.¹

The customer and the calibration laboratory shall agree on the details of the tests for an individual calibration, considering the normal use of the instrument. The parties shall also agree on additional tests that may support the performance evaluation of the instrument under special conditions of use. Such agreement should be consistent with the following sections.

8.1 Indication

According to the method presented in this document, the no-load indications and the loaded indications are recorded for the selected calibration points.

The indication i related to a test load, is the difference of the indication with load i_L and the average \bar{i}_0 of the indications without load recorded before i_{0_1} and after i_{0_2} of the test load:

$$i = i_L - \bar{i}_0 \quad (8.1-1)$$

with,

$$\bar{i}_0 = \frac{i_{0_1} + i_{0_2}}{2} \quad (8.1-2)$$

The instrument indication at any test load, or no load, is read and recorded only if it can be considered as stable. Otherwise, an average value of the indication should be documented along with information about the observed variability. During calibration tests, the original indications should be recorded, not the errors or variations in the indication.

¹ Adequate implementation of this measurement method minimizes the effect of eccentric load. If it is necessary to evaluate the eccentric loading effect, see [2].

8.2 Description of the method

Calibration of non-automatic weighing instruments with a resolution of less than 0.010 mg requires a reference weight and a set of auxiliary weights of intermediate values to determine the error of the corresponding indication at the selected points for the error of indication test.

The method consists of performing a series of 31 weighing cycles as follows:

- a) Define 10 points in the selected calibration range including the nominal value of the standard weight. If possible, select test loads equidistant from each other.
- b) The first and last weighing cycles correspond to the reference weight.
- c) For each test load selected, three weighing cycles are performed, except for the minimum load (Min') where two weighing cycles are performed.
- d) Each weighing cycle is performed following the sequence $i_{0_1} \rightarrow i_L \rightarrow i_{0_2}$, using a different combination of auxiliary weights.
- e) Auxiliary weights should be combined in such a way as to ensure the use of each weight at least twice in the complete measurement series.
- f) The environmental conditions (temperature, relative humidity and atmospheric pressure) should be recorded at the beginning and end of the series, and each time there is a change in the test load.
- g) Adjustment of the instrument should be performed at the beginning of the series and each time there is a change in the test load.

Figure 1 shows an example of the sequence described above.

Figure 1. Measurement sequence

Test points	Zero (no-load)	Load	Zero (no-load)
Record environmental conditions			
Perform adjustment			
Reference weight			
1st test point			
1st test point			
Record environmental conditions			
Perform adjustment			
2nd test point			
2nd test point			
2nd test point			
Record environmental conditions			
Perform adjustment			
⋮			
10th test point			
10th test point			
10th test point			
Record environmental conditions			
Perform adjustment			
Reference weight			

8.2.1 Operation routine.

The operation routine for a complete weighing series is shown below:

Record environmental conditions

Perform adjustment

Record reference weight

1st Test Point

- Record the first combination of auxiliary weights.
- Record the second combination of auxiliary weights.
- Record environmental conditions

2nd to 10th Test Point

- Perform adjustment
- Record the first combination of auxiliary weights.
- Record the second combination of auxiliary weights.
- Record the third combination of auxiliary weights.
- Record environmental conditions

Perform adjustment

Record reference weight

Repeat the operation routine 3 times (i.e. 3 measurement series).

9 MEASUREMENT RESULTS

The measurement method used in the calibration non-automatic weighing instruments with a resolution of less than 0.010 mg makes it possible to determine the error of indication and the calibration uncertainty of the measuring instrument under test. The implementation of this method allows obtaining uncertainties lower than those obtained under the methodology described in SIM Guide MWG7/cg-01 [2].

9.1 Measurement model

From section 5.2, when placing a certain combination of auxiliary weights, the indication of the instrument can be expressed in a general way as:

$$i_i = m_{N_i} + \delta m_i + e_i + b_i \quad (9.1-1)$$

Where:

- i_i is the i -th indication of the instrument, for $i = 2, \dots, m - 1$
- $m_{N_i} + \delta m_i$ is the conventional mass of the combination of auxiliary weights², with nominal value m_N and correction δm_i for $i = 2, \dots, m - 1$ used to obtain the i -th indication of the instrument.
- e_i is the error of indication of the instrument at i -th test point for $i = 2, \dots, m - 1$
- b_i is air buoyancy correction of the auxiliary weights combination for $i = 2, \dots, m - 1$

Particularly, for the case of the indication of the reference weight, which is measured at the beginning and at the end according to the scheme shown in Figure 1, we have:

$$i_i = m_{N_R} + \delta m_R + e_i + b_i \quad (9.1-2)$$

Where:

- i_i is the i -th indication of the instrument, for $i = 1, i = m$
- m_{N_R} is the nominal value of the reference weight
- δm_R is the conventional mass correction of the weight
- e_i is the indication error of the instrument, for $i = 1, i = m$

² $m_{N_i} + \delta m_i$ generally represents the conventional mass of the auxiliary weights, whether one or a combination of two or more. If more than one weight is used, $\sum(m_{N_i} + \delta m_i)$ applies.

b_i is the air buoyancy correction of the reference weight for $i = 1, i = m$

The air buoyancy correction is generally calculated with the following equation:

$$b_i = -m_{N_i}(\rho_a - \rho_0)(1/\rho_i - 1/\rho_c) \quad (9.1-3a)$$

Where:

- b_i is the air buoyancy correction for the i -th measurement
- ρ_a is the air density during calibration
- ρ_0 is the conventional air density equal to 0.0012 g cm^{-3}
- ρ_i is the density of the weights used in the i -th measurement³
- ρ_c is the conventional density of stainless steel equal to 8.0 g cm^{-3}

Equation (9.1-3a) can be rewritten in terms of the volume V_i of the weights used as follows:

$$b_i = -(\rho_a - \rho_0)(V_i - m_{N_i}/\rho_c) \quad (9.1-3b)$$

Expressions (9.1-1) and (9.1-2) can be rewritten as:

$$i_i - \sum(m_{N_i}) = \sum(\delta m_i) + e_i + b_i \quad \text{for } i = 2, \dots, m-1 \quad (9.1-1a)$$

$$i_i - m_{N_R} = \delta m_R + e_i + b_i \quad \text{for } i = 1, i = m \quad (9.1-2a)$$

Equations (9.1-1a) and (9.1-2a) can be expressed in a matrix form through a design matrix $\mathbf{A}_{m \times n}$, whose elements are zeros (0) and ones (1). Each row represents the measurement scheme of Table 1 including the combinations of auxiliary weights used, the errors of indication and the corrections of the conventional masses of the auxiliary weights. Additionally, in the first and last row, the known value of the conventional mass of the reference weight is added. The matrix expression is:

$$\mathbf{I} = \mathbf{A}\mathbf{E} + \delta m_R \mathbf{P} + \mathbf{B} \quad (9.1-4)$$

Where:

- \mathbf{I} is the column vector $m \times 1$ containing the measurements minus the nominal value of the test point.
- \mathbf{A} is the design matrix $m \times n$ whose elements are zeros (0) and ones (1).

³ The density of a set of weights can be obtained using equation B.7.9-1 of OIML R111-1:2004 [5].

- E*** is the column vector $n \times 1$ of the unknowns to be estimated, which are the indication errors of the balance and the conventional mass corrections of the auxiliary weights.
- P*** is a column vector $m \times 1$ whose initial and final element values are equal to one (1) and the others are zeros (0).
- B*** is the column vector $m \times 1$ of the air buoyancy corrections.

The matrix equation (9.1-4) can be written as:

$$\begin{aligned} I - \delta m_R P - B &= AE \\ Y &= AE + R \end{aligned} \tag{9.1-5}$$

With $Y = I - \delta m_R P - B$

The matrix equation (9.1-5) can be solved by ordinary least squares fitting to determine the vector of estimates ***E***.

The vector ***R*** is the column vector $m \times 1$ of the residuals due to the fit.

The least-squares solution of equation (9.1-5) is given by:

$$E = (A^T A)^{-1} A^T Y \tag{9.1-6}$$

The values of ***R*** have mean value 0 and variance ***Uresid*** so that the expression (9.1-5) has a solution by applying the least squares method and obtaining the equation (9.1-6).

10 UNCERTAINTY CALCULATION OF THE ESTIMATES OF ERRORS OF INDICATION AND CONVENTIONAL MASS CORRECTIONS OF AUXILIARY WEIGHTS

From equation (9.1-6), the propagation of uncertainty of E is obtained with:

$$\mathbf{U}_E = [(\mathbf{A}^T \mathbf{A})^{-1} \mathbf{A}^T] \mathbf{U}_Y [\mathbf{A} (\mathbf{A}^T \mathbf{A})^{-1}] \quad (10-1)$$

\mathbf{U}_E is the covariance matrix $n \times n$ that contains, on its diagonal, the variances in the estimates of E , and off the diagonal, the corresponding covariances. Therefore, the standard uncertainty associated with the estimates of E , is obtained by calculating the square root of the elements of the diagonal of \mathbf{U}_E . That is:

$$u_{E_i} = \sqrt{(\mathbf{U}_E)_{ij}} \quad \text{para } i = j \quad (10-2)$$

\mathbf{U}_Y is the covariance matrix (of dimensions $m \times m$) associated with the uncertainty contribution of the measurement process. This matrix is constructed with the covariance matrices due to

- repeatability of measurements, \mathbf{U}_{rep}
- the residuals due to the least squares adjustment $\mathbf{U}_{\text{resid}}$
- the resolution of the instrument, \mathbf{U}_{res}
- correction for air buoyancy, \mathbf{U}_b
- the reference weight. \mathbf{U}_{m_R}

Symbolically:

$$\mathbf{U}_Y = \mathbf{U}_{\text{rep}} + \mathbf{U}_{\text{res}} + \mathbf{U}_{\text{resid}} + \mathbf{U}_b + \mathbf{U}_{m_R} \quad (10-3)$$

It follows from the above that all covariance matrices that make up \mathbf{U}_Y have dimension $m \times m$.

10.1 Matrix \mathbf{U}_{rep}

It is made up with the variance due to the repeatability $u_{\text{rep}_i}^2$ of the measurement series for each of the test points. For $k = 1, 2, \dots, N$ number of series, the standard uncertainty due to this component at the i -th test point is:

$$u_{\text{rep}_i} = \frac{s_i}{\sqrt{N}} \quad (10.1-1)$$

With:

$$s_i = \sqrt{\frac{1}{N-1} \sum_{k=1}^N (i_{ki} - \bar{i}_i)^2} \quad (10.1-2)$$

The covariance matrix \mathbf{U}_{rep} is a square matrix $m \times m$ whose diagonal elements are the variances $u_{\text{rep}_i}^2$ and the off-diagonal elements are equal to zero, i.e:

$$(\mathbf{U}_{\text{rep}})_{ij} = \begin{cases} u_{\text{rep}_i}^2 & \text{para } i = j \\ 0 & \text{para } i \neq j \end{cases} \quad (10.1-3)$$

In matrix form:

$$\mathbf{U}_{\text{rep}_{m \times m}} = \begin{vmatrix} u_{\text{rep}_1}^2 & 0 & \dots & 0 & 0 \\ 0 & u_{\text{rep}_2}^2 & \dots & 0 & 0 \\ \vdots & 0 & \ddots & 0 & \vdots \\ 0 & 0 & \dots & u_{\text{rep}_{m-1}}^2 & 0 \\ 0 & 0 & \dots & 0 & u_{\text{rep}_m}^2 \end{vmatrix}$$

10.2 Matrix \mathbf{U}_{res}

It is made up with the variance u_{res}^2 of the resolution d of the instrument under calibration. Therefore:

$$u_{\text{res}} = \frac{d}{2\sqrt{3}} \sqrt{2} \quad (10.2-1)$$

The term $\sqrt{2}$ is due to the fact that the indication of the balance is corrected by zero, therefore, the resolution of the instrument with and without load is taken into account.

The covariance matrix \mathbf{U}_{res} is equal to:

$$\mathbf{U}_{\text{res}} = u_{\text{res}}^2 \mathbf{I}_d \quad (10.2-2)$$

Where I_d is the identity matrix with dimension $m \times m$.

In matrix form:

$$U_{\text{res}m \times m} = \begin{vmatrix} u_{\text{res}}^2 & 0 & \cdots & 0 & 0 \\ 0 & u_{\text{res}}^2 & \cdots & 0 & 0 \\ \vdots & 0 & \ddots & 0 & \vdots \\ 0 & 0 & \cdots & u_{\text{res}}^2 & 0 \\ 0 & 0 & \cdots & 0 & u_{\text{res}}^2 \end{vmatrix}$$

10.3 Matrix U_{resid}

It is made up with the variance due to the residuals of the ordinary least squares fitting, u_{resid}^2 . This variance is obtained as the square of the error of the residuals $R = Y - AE$ among the degrees of freedom of the fitting. Therefore:

$$u_{\text{resid}}^2 = \frac{(Y-AE)^T(Y-AE)}{m-n} \quad (10.3-1)$$

Where:

- m is the number of rows in the design matrix A .
- n is the number of columns of the design matrix A .

The covariance matrix U_{resid} is equal to:

$$U_{\text{resid}} = u_{\text{resid}}^2 I_d \quad (10.3-2)$$

I_d is the identity matrix with dimension $m \times m$.

In matrix form:

$$U_{\text{resid}m \times m} = \begin{vmatrix} u_{\text{resid}}^2 & 0 & \cdots & 0 & 0 \\ 0 & u_{\text{resid}}^2 & \cdots & 0 & 0 \\ \vdots & 0 & \ddots & 0 & \vdots \\ 0 & 0 & \cdots & u_{\text{resid}}^2 & 0 \\ 0 & 0 & \cdots & 0 & u_{\text{resid}}^2 \end{vmatrix}$$

10.4 Matrix U_b

It is made up with the variance due to the air buoyancy correction $u_{b_i}^2$, for each i -th measurement. Applying GUM [1] to equations (9.1-3a) with the density of the weights and (9.1-3b) with the volume of the weights, we obtain:

$$u_{b_i}^2 = m_{N_i}^2 (1/\rho_i - 1/\rho_c)^2 u_{\rho_a}^2 + m_{N_i}^2 (\rho_a - \rho_0)^2 \frac{u_{\rho_i}^2}{\rho_i^4} \quad (10.4-1a)$$

$$u_{b_i}^2 = (V_i - m_{N_i}/\rho_c)^2 u_{\rho_a}^2 + (\rho_a - \rho_0)^2 u_{V_i}^2 \quad (10.4-1b)$$

Where:

- u_{b_i} is the standard uncertainty due to air buoyancy correction for the i -th measurement,
- u_{ρ_a} is the standard uncertainty due to air density during calibration,
- u_{ρ_i} is the density of the weights used in the i -th measurement in equation (10.4-1a)
- u_{V_i} is the volume of the weights used in the i -th measurement in equation (10.4-1b)

The covariance matrix U_b is a square matrix $m \times m$ whose diagonal elements are the variances $u_{b_i}^2$ and the off-diagonal elements are equal to zero, i.e:

$$(U_b)_{ij} = \begin{cases} u_{b_i}^2 & \text{para } i = j \\ 0 & \text{para } i \neq j \end{cases} \quad (10.4-2)$$

In matrix form:

$$U_{b_{m \times m}} = \begin{vmatrix} u_{b_1}^2 & 0 & \dots & 0 & 0 \\ 0 & u_{b_2}^2 & \dots & 0 & 0 \\ \vdots & 0 & \ddots & 0 & \vdots \\ 0 & 0 & \dots & u_{b_{m-1}}^2 & 0 \\ 0 & 0 & \dots & 0 & u_{b_m}^2 \end{vmatrix}$$

10.5 Matrix U_{m_R}

It is made up with the variance due to the conventional mass correction δm_R of the reference weight $u_{m_R}^2$, where:

$$u_{m_R}^2 = u_{cal}^2 + u_{inst}^2 \quad (10.5-1)$$

With:

u_{cal} standard uncertainty of the calibration of the reference weight,
 u_{inst} standard uncertainty due to the instability of the reference weight, which can be obtained from the history of its successive calibrations.

According to equation (9.1-5) the covariance matrix U_{m_R} is equal to:

$$U_{m_R} = u_{m_R}^2 \mathbf{P}\mathbf{P}^T \quad (10.5-2)$$

The product $\mathbf{P}\mathbf{P}^T$ results in a matrix $m \times m$ where the elements of its corners contain $u_{m_R}^2$, and the other elements are equal to zero, that is:

$$(U_{m_R})_{1,1} = (U_{m_R})_{m,1} = (U_{m_R})_{1,m} = (U_{m_R})_{m,m} = u_{m_R}^2$$

In matrix form this is:

$$U_{m_R m \times m} = \begin{vmatrix} u_{m_R}^2 & 0 & \cdots & 0 & u_{m_R}^2 \\ 0 & 0 & \cdots & 0 & 0 \\ \vdots & 0 & \ddots & 0 & \vdots \\ 0 & 0 & \cdots & 0 & 0 \\ u_{m_R}^2 & 0 & \cdots & 0 & u_{m_R}^2 \end{vmatrix}$$

After having calculated each component of U_Y , U_E is calculated with equation (10-1). Finally, the standard uncertainty associated with the estimates is obtained with equation (10-2).

11 TRACEABILITY OF MEASUREMENTS

11.1 Good measurement practices

In order to perform calibrations and mass measurements reliably and within the usual working range of weighing instruments with a resolution of less than 0.010 mg, some recommendations are presented below:

- The weight used as reference standard must have its mass calibration certificate.
- Both the reference weight and auxiliary weights must have their volume or density value, preferably with a calibration certificate. The volume or density can be measured using any of the methods set out in section B.7 of OIML R111:2004 [5] depending on the required uncertainty.
- Instruments or auxiliary equipment used for measuring environmental conditions must have their corresponding calibration certificate.
- Calibration certificates for all instruments, equipment and standards used in calibration must follow the calibration program established by the laboratory.
- Maintain environmental conditions within the appropriate working values.
- Establish measures to control the entry of dust into the calibration area.
- Whenever any type of maintenance is carried out on the instrument that significantly affects the result of the mass measurements made with it, the instrument must be adjusted and calibrated afterwards.
- Laboratory personnel or the user should ensure that the instrument is properly installed prior to use.
- During cleaning of the weighing pan or load receptor of the instrument, dust residues (or any other substance) must be removed.
- All instrument indications should be recorded only when the instrument has reached stability. The laboratory should have a criterion for determining the stabilization time of the indications and when stable, record the corresponding indications.
- The laboratory should include in its procedures the relevant activities to be performed during calibration.
- Perform calibration and proper storage of the adjustment weight (for external adjustment option only) so that its physical and metrological characteristics are not contaminated or affected.

12 REFERENCES

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13 APPENDICES

13.1 APPENDIX A

13.1.1 Appendix A1: EXAMPLE

13.1.1.1 Initial information.

Calibration of the instrument for weighing with $Max = 5 \text{ g} / d = 0.1 \text{ }\mu\text{g}$.

Calibration interval: From $Min' = 500 \text{ mg}$ to $Max = 5 \text{ g}$

The reference weight selected is the one with a nominal value equal or close to the maximum value of the calibration range. In this case, a recently calibrated 5 g weight of accuracy class E₁ is used. The data of the reference weight are:

ID	Conventional Mass m_R	$U_{m_R} (k = 2)$	Volume V_R	$U_{V_R} (k = 2)$
m_R	5 g + 0.244 mg	0.005 mg	0.629 3 cm ³	0.001 5 cm ³

According to 7.2, the test points for the selected span calibration are (10 points equidistant from the calibration span):

	1	2	3	4	5	6	7	8	9	10
Test Point	0.5 g	1.0 g	1.5 g	2.0 g	2.5 g	3.0 g	3.5 g	4.0 g	4.5 g	5.0 g

The auxiliary weights required for the selected test points are:

ID	Nominal value	Volume V_i	$U_{V_i} (k = 2)$
$m_{0.5}$	0.5 g	0.063 0 cm ³	0.000 8 cm ³
$m_{0.5*}$	0.5 g	0.063 0 cm ³	0.000 8 cm ³
m_1	1 g	0.125 7 cm ³	0.000 8 cm ³
m_{1*}	1 g	0.125 7 cm ³	0.000 9 cm ³
m_2	2 g	0.251 5 cm ³	0.000 8 cm ³
m_{2*}	2 g	0.251 3 cm ³	0.001 2 cm ³

From the above and following the criteria of 7.2, the measurement scheme chosen for the calibration of the instrument span is as follows:

Table 2. Measurement scheme for instrument calibration.

No.	Load	Weights / g						
	g	$m_{0.5}$	$m_{0.5^*}$	m_1	m_{1^*}	m_2	m_{2^*}	m_5
		Temp / °C =		r.h. / % =		Pressure / hPa =		
1	5.0	0	0	0	0	0	0	1
2	0.5	1	0	0	0	0	0	0
3	0.5	0	1	0	0	0	0	0
		Temp / °C =		r.h. / % =		Pressure / hPa =		
4	1.0	1	1	0	0	0	0	0
5	1.0	0	0	1	0	0	0	0
6	1.0	0	0	0	1	0	0	0
		Temp / °C =		r.h. / % / h.r.		Pressure / hPa =		
7	1.5	1	0	1	0	0	0	0
8	1.5	0	1	0	1	0	0	0
9	1.5	1	0	0	1	0	0	0
		Temp / °C =		r.h. / % =		Pressure / hPa =		
10	2.0	0	0	0	0	1	0	0
11	2.0	0	0	0	0	0	1	0
12	2.0	0	0	1	1	0	0	0
		Temp / °C =		r.h. / % =		Pressure / hPa =		
13	2.5	1	0	0	0	1	0	0
14	2.5	0	1	0	0	0	1	0
15	2.5	0	1	1	1	0	0	0
		Temp / °C =		r.h. / % =		Pressure / hPa =		
16	3.0	0	0	1	0	1	0	0
17	3.0	0	0	0	1	0	1	0
18	3.0	1	1	0	0	1	0	0
		Temp / °C =		r.h. / % =		Pressure / hPa =		
19	3.5	1	0	1	0	1	0	0
20	3.5	0	1	0	0	0	1	0
21	3.5	0	1	0	1	1	0	0
		Temp / °C =		r.h. / % =		Pressure / hPa =		
22	4.0	0	0	0	0	1	1	0
23	4.0	0	0	1	1	1	0	0
24	4.0	1	1	1	0	0	1	0
		Temp / °C =		r.h. / % =		Pressure / hPa =		
25	4.5	1	0	1	1	0	1	0
26	4.5	0	1	1	1	0	1	0
27	4.5	1	0	0	0	1	1	0
		Temp / °C =		r.h. / % =		Pressure / hPa =		
28	5.0	0	0	1	0	1	1	0
29	5.0	0	0	0	1	1	1	0
30	5.0	1	1	0	0	1	1	0
		Temp / °C =		r.h. / % =		Pressure / hPa =		
31	5.0	0	0	0	0	0	0	1

13.1.1.2 Measurements.

Based on Table 2, the indications of three measurement series are recorded:

Series 1:

No.	Load	Weights / g							Indications for microbalance / mg		
	g	$m_{0.5}$	$m_{0.5^*}$	m_1	m_{1^*}	m_2	m_{2^*}	m_5	Zero	Cargo	Zero
		Temp / °C =	20.2	r.h. / % =	50.8	Pressure / hPa =	752.7	<i>Sensitivity adjustment</i>			
1	5.0	0	0	0	0	0	0	1	0.000 0	5 000.246 1	-0.000 2
2	0.5	1	0	0	0	0	0	0	0.000 0	500.455 8	0.0 00 8
3	0.5	0	1	0	0	0	0	0	0.000 0	500.300 6	0.000 2
		Temp / °C =	20.0	r.h. / % =	51.5	Pressure / hPa =	752.7	<i>Sensitivity adjustment</i>			
4	1.0	1	1	0	0	0	0	0	0.000 0	1 000.757 1	0.000 4
5	1.0	0	0	1	0	0	0	0	0.000 0	999.766 0	0.000 0
6	1.0	0	0	0	1	0	0	0	0.000 0	999.890 9	0.000 3
		Temp / °C =	19.9	r.h. / % / h.r.	52.2	Pressure / hPa =	752.8	<i>Sensitivity adjustment</i>			
7	1.5	1	0	1	0	0	0	0	0.000 0	1 500.222 2	0.000 6
8	1.5	0	1	0	1	0	0	0	0.000 0	1 500.191 9	0.000 6
9	1.5	1	0	0	1	0	0	0	0.000 0	1 500.346 1	-0.000 1
		Temp / °C =	19.9	r.h. / % =	52.2	Pressure / hPa =	752.8	<i>Sensitivity adjustment</i>			
10	2.0	0	0	0	0	1	0	0	0.000 0	1 999.397 8	0.000 3
11	2.0	0	0	0	0	0	1	0	0.000 0	1 999.862 9	0.000 2
12	2.0	0	0	1	1	0	0	0	0.000 0	1 999.656 3	0.000 3
		Temp / °C =	19.9	r.h. / % =	52.3	Pressure / hPa =	752.8	<i>Sensitivity adjustment</i>			
13	2.5	1	0	0	0	1	0	0	0.000 0	2 499.853 8	0.001 2
14	2.5	0	1	0	0	0	1	0	0.000 0	2 500.164 4	-0.000 1
15	2.5	0	1	1	1	0	0	0	0.000 0	2 499.958 5	0.000 4
		Temp / °C =	19.9	r.h. / % =	52.5	Pressure / hPa =	752.8	<i>Sensitivity adjustment</i>			
16	3.0	0	0	1	0	1	0	0	0.000 0	2 999.162 4	0.000 6
17	3.0	0	0	0	1	0	1	0	0.000 0	2 999.753 0	0.000 7
18	3.0	1	1	0	0	1	0	0	0.000 0	3 000.152 6	0.000 2
		Temp / °C =	19.9	r.h. / % =	52.4	Pressure / hPa =	752.9	<i>Sensitivity adjustment</i>			
19	3.5	1	0	1	0	1	0	0	0.000 0	3 499.620 0	0.000 1
20	3.5	0	1	0	1	0	1	0	0.000 0	3 500.054 5	0.000 7
21	3.5	0	1	0	1	1	0	0	0.000 0	3 499.588 0	0.000 0
		Temp / °C =	19.9	r.h. / % =	52.2	Pressure / hPa =	752.8	<i>Sensitivity adjustment</i>			
22	4.0	0	0	0	0	1	1	0	0.000 0	3 999.259 4	0.000 7
23	4.0	0	0	1	1	1	0	0	0.000 0	3 999.053 3	0.000 3
24	4.0	1	1	1	0	0	1	0	0.000 0	4 000.384 3	0.000 4
		Temp / °C =	19.8	r.h. / % =	52.0	Pressure / hPa =	752.8	<i>Sensitivity adjustment</i>			
25	4.5	1	0	1	1	0	1	0	0.000 0	4 499.975 6	0.001 1
26	4.5	0	1	1	1	0	1	0	0.000 0	4 499.819 0	0.000 3
27	4.5	1	0	0	0	1	1	0	0.000 0	4 499.714 1	0.000 4
		Temp / °C =	19.9	r.h. / % =	51.8	Pressure / hPa =	753.0	<i>Sensitivity adjustment</i>			
28	5.0	0	0	1	0	1	1	0	0.000 0	4 999.026 0	0.000 5
29	5.0	0	0	0	1	1	1	0	0.000 0	4 999.148 3	-0.000 1
30	5.0	1	1	0	0	1	1	0	0.000 0	5 000.015 7	0.000 3
		Temp / °C =	19.9	r.h. / % =	51.7	Pressure / hPa =	753.0	<i>Sensitivity adjustment</i>			
31	5.0	0	0	0	0	0	0	1	0.000 0	5 000.248 0	0.000 9

SIM Guidelines on the calibration of non-automatic weighing instruments with resolution less than 0.010 mg

Series 2:

No.	Load	Weights / g							Indications for microbalance / mg		
	g	$m_{0.5}$	$m_{0.5^*}$	m_1	m_{1^*}	m_2	m_{2^*}	m_5	Zero	Cargo	Zero
		Temp / °C =	19.7	r.h. / % =	52.3	Pressure / hPa =	752.4	Sensitivity adjustment			
1	5.0	0	0	0	0	0	0	1	0.000 0	5 000.247 8	0.000 7
2	0.5	1	0	0	0	0	0	0	0.000 0	500.455 0	0.000 0
3	0.5	0	1	0	0	0	0	0	0.000 0	500.299 7	-0.000 4
		Temp / °C =	19.8	r.h. / % =	52.4	Pressure / hPa =	752.4	Sensitivity adjustment			
4	1.0	1	1	0	0	0	0	0	0.000 0	1 000.755 7	0.000 1
5	1.0	0	0	1	0	0	0	0	0.000 0	999.766	-0.000 1
6	1.0	0	0	0	1	0	0	0	0.000 0	999.890	-0.000 1
		Temp / °C =	19.8	r.h. / % / h.r.	52.5	Pressure / hPa =	752.4	Sensitivity adjustment			
7	1.5	1	0	1	0	0	0	0	0.000 0	1 500.222 4	0.000 9
8	1.5	0	1	0	1	0	0	0	0.000 0	1 500.190 6	-0.000 2
9	1.5	1	0	0	1	0	0	0	0.000 0	1 500.345 6	0.000 3
		Temp / °C =	19.8	r.h. / % =	52.4	Pressure / hPa =	752.4	Sensitivity adjustment			
10	2.0	0	0	0	0	1	0	0	0.000 0	1 999.399 0	0.000 6
11	2.0	0	0	0	0	0	1	0	0.000 0	1 999.863 2	0.000 2
12	2.0	0	0	1	1	0	0	0	0.000 0	1 999.657 1	0.000 4
		Temp / °C =	19.8	r.h. / % =	52.5	Pressure / hPa =	752.4	Sensitivity adjustment			
13	2.5	1	0	0	0	1	0	0	0.000 0	2 499.853 1	0.000 7
14	2.5	0	1	0	0	0	1	0	0.000 0	2 500.164 1	0.000 2
15	2.5	0	1	1	1	0	0	0	0.000 0	2 499.955 8	0.000 2
		Temp / °C =	19.8	r.h. / % =	52.3	Pressure / hPa =	752.3	Sensitivity adjustment			
16	3.0	0	0	1	0	1	0	0	0.000 0	2 999.162 8	0.000 7
17	3.0	0	0	0	1	0	1	0	0.000 0	2 999.753 0	0.000 3
18	3.0	1	1	0	0	1	0	0	0.000 0	3 000.153 3	-0.000 1
		Temp / °C =	19.8	r.h. / % =	52.4	Pressure / hPa =	752.2	Sensitivity adjustment			
19	3.5	1	0	1	0	1	0	0	0.000 0	3 499.619 3	0.000 9
20	3.5	0	1	0	1	0	1	0	0.000 0	3 500.053 7	0.000 2
21	3.5	0	1	0	1	1	0	0	0.000 0	3 499.587 4	-0.000 1
		Temp / °C =	19.8	r.h. / % =	52.0	Pressure / hPa =	752.2	Sensitivity adjustment			
22	4.0	0	0	0	0	1	1	0	0.000 0	3 999.258 2	0.000 7
23	4.0	0	0	1	1	1	0	0	0.000 0	3 999.053 4	0.000 3
24	4.0	1	1	1	0	0	1	0	0.000 0	4 000.384 5	0.000 2
		Temp / °C =	19.8	r.h. / % =	51.9	Pressure / hPa =	752.1	Sensitivity adjustment			
25	4.5	1	0	1	1	0	1	0	0.000 0	4 499.975 4	0.001 1
26	4.5	0	1	1	1	0	1	0	0.000 0	4 499.819 9	-0.000 2
27	4.5	1	0	0	0	1	1	0	0.000 0	4 499.715 1	0.000 1
		Temp / °C =	19.9	r.h. / % =	52.1	Pressure / hPa =	752.1	Sensitivity adjustment			
28	5.0	0	0	1	0	1	1	0	0.000 0	4 999.026 2	0.000 7
29	5.0	0	0	0	1	1	1	0	0.000 0	4 999.148 9	0.000 1
30	5.0	1	1	0	0	1	1	0	0.000 0	5 000.014 1	0.000 0
		Temp / °C =	19.9	r.h. / % =	52.0	Pressure / hPa =	752.1	Sensitivity adjustment			
31	5.0	0	0	0	0	0	0	1	0.000 0	5 000.248 8	0.000 9

Series 3:

No.	Load	Weights / g							Indications for microbalance / mg		
	g	$m_{0.5}$	$m_{0.5^*}$	m_1	m_{1^*}	m_2	m_{2^*}	m_5	Zero	Cargo	Zero
		Temp / °C =	19.7	r.h. / % =	52.3	Pressure / hPa =	752.4	Sensitivity adjustment			
1	5.0	0	0	0	0	0	0	1	0.000 0	5000.247 8	0.000 7
2	0.5	1	0	0	0	0	0	0	0.000 0	500.455 0	0.000 0
3	0.5	0	1	0	0	0	0	0	0.000 0	500.299 7	-0.000 4
		Temp / °C =	19.8	r.h. / % =	52.4	Pressure / hPa =	752.4	Sensitivity adjustment			
4	1.0	1	1	0	0	0	0	0	0.000 0	1 000.755 7	0.000 1
5	1.0	0	0	1	0	0	0	0	0.000 0	999.766 4	-0.000 1
6	1.0	0	0	0	1	0	0	0	0.000 0	999.890 1	-0.000 1
		Temp / °C =	19.8	r.h. / % / h.r.	52.5	Pressure / hPa =	752.4	Sensitivity adjustment			
7	1.5	1	0	1	0	0	0	0	0.000 0	1 500.222 4	0.000 9
8	1.5	0	1	0	1	0	0	0	0.000 0	1 500.190 6	-0.000 2
9	1.5	1	0	0	1	0	0	0	0.000 0	1 500.345 6	0.000 3
		Temp / °C =	19.8	r.h. / % =	52.4	Pressure / hPa =	752.4	Sensitivity adjustment			
10	2.0	0	0	0	0	1	0	0	0.000 0	1 999.399 0	0.000 6
11	2.0	0	0	0	0	0	1	0	0.000 0	1 999.863 2	0.000 2
12	2.0	0	0	1	1	0	0	0	0.000 0	1 999.657 1	0.000 4
		Temp / °C =	19.8	r.h. / % =	52.5	Pressure / hPa =	752.4	Sensitivity adjustment			
13	2.5	1	0	0	0	1	0	0	0.000 0	2 499.853 1	0.000 7
14	2.5	0	1	0	0	0	1	0	0.000 0	2 500.164 1	0.000 2
15	2.5	0	1	1	1	0	0	0	0.000 0	2 499.955 8	0.000 2
		Temp / °C =	19.8	r.h. / % =	52.3	Pressure / hPa =	752.3	Sensitivity adjustment			
16	3.0	0	0	1	0	1	0	0	0.000 0	2 999.162 8	0.000 7
17	3.0	0	0	0	1	0	1	0	0.000 0	2 999.753 0	0.000 3
18	3.0	1	1	0	0	1	0	0	0.000 0	3 000.153 3	-0.000 1
		Temp / °C =	19.8	r.h. / % =	52.4	Pressure / hPa =	752.2	Sensitivity adjustment			
19	3.5	1	0	1	0	1	0	0	0.000 0	3 499.619 3	0.000 9
20	3.5	0	1	0	1	0	1	0	0.000 0	3 500.053 7	0.000 2
21	3.5	0	1	0	1	1	0	0	0.000 0	3 499.587 4	-0.000 1
		Temp / °C =	19.8	r.h. / % =	52.0	Pressure / hPa =	752.2	Sensitivity adjustment			
22	4.0	0	0	0	0	1	1	0	0.000 0	3 999.258 2	0.000 7
23	4.0	0	0	1	1	1	0	0	0.000 0	3 999.053 4	0.000 3
24	4.0	1	1	1	0	0	1	0	0.000 0	4 000.384 5	0.000 2
		Temp / °C =	19.8	r.h. / % =	51.9	Pressure / hPa =	752.1	Sensitivity adjustment			
25	4.5	1	0	1	1	0	1	0	0.000 0	4 499.975 4	0.001 1
26	4.5	0	1	1	1	0	1	0	0.000 0	4 499.819 9	-0.000 2
27	4.5	1	0	0	0	1	1	0	0.000 0	4 499.715 1	0.000 1
		Temp / °C =	19.9	r.h. / % =	52.1	Pressure / hPa =	752.1	Sensitivity adjustment			
28	5.0	0	0	1	0	1	1	0	0.000 0	4 999.026 2	0.000 7
29	5.0	0	0	0	1	1	1	0	0.000 0	4 999.148 9	0.000 1
30	5.0	1	1	0	0	1	1	0	0.000 0	5 000.014 1	0.000 0
		Temp / °C =	19.9	r.h. / % =	52.0	Pressure / hPa =	752.1	Sensitivity adjustment			
31	5.0	0	0	0	0	0	0	1	0.000 0	5 000.248 8	0.000 9

13.1.1.3 Calculation of estimates.

For each row of each measurement series, the zero-corrected indications are obtained with equations (8.1-1) and (8.1-2). Subsequently, the errors of indication for each row are obtained with the difference of the zero-corrected indication minus the nominal value of the test load, i.e., $e_i = i_i - m_{N_i}$:

No.	Load g	Indications Corrected by zero / mg			Errors of Indication / mg		
		Series 1	Series 2	Series 3	Series 1	Series 2	Series 3
1	5.0	5000.246 2	5 000.247 5	5 000.247 4	0.246 20	0.247 45	0.247 35
2	0.5	500.455 4	500.455 0	500.455 9	0.455 40	0.455 00	0.455 85
3	0.5	500.300 5	500.299 9	500.300 9	0.300 50	0.299 90	0.300 90
4	1.0	1 000.756 9	1 000.755 7	1 000.755 9	0.756 90	0.755 65	0.755 85
5	1.0	999.766 0	999.766 5	999.765 9	-0.234 00	-0.233 55	-0.234 10
6	1.0	999.890 8	999.890 2	999.891 4	-0.109 25	-0.109 85	-0.108 65
7	1.5	1 500.221 9	1 500.222 0	1 500.222 4	0.221 90	0.221 95	0.222 35
8	1.5	1 500.191 6	1 500.190 7	1 500.191 9	0.191 60	0.190 70	0.191 85
9	1.5	1 500.346 2	1 500.345 5	1 500.347 2	0.346 15	0.345 45	0.347 20
10	2.0	1 999.397 7	1 999.398 7	1 999.398 1	-0.602 35	-0.601 30	-0.601 90
11	2.0	1 999.862 8	1 999.863 1	1 999.863 2	-0.137 20	-0.136 90	-0.136 85
12	2.0	1 999.656 2	1 999.656 9	1 999.656 2	-0.343 85	-0.343 10	-0.343 85
13	2.5	2 499.853 2	2 499.852 8	2 499.853 9	-0.146 80	-0.147 25	-0.146 10
14	2.5	2 500.164 5	2 500.164 0	2 500.164 1	0.164 45	0.164 00	0.164 05
15	2.5	2 499.958 3	2 499.955 7	2 499.957 5	-0.041 70	-0.044 30	-0.042 50
16	3.0	2 999.162 1	2 999.162 5	2 999.162 2	-0.837 90	-0.837 55	-0.837 80
17	3.0	2 999.752 7	2 999.752 9	2 999.752 7	-0.247 35	-0.247 15	-0.247 35
18	3.0	3 000.152 5	3 000.153 4	3 000.153 3	0.152 50	0.153 35	0.153 25
19	3.5	3 499.620 0	3 499.618 9	3 499.619 1	-0.380 05	-0.381 15	-0.380 95
20	3.5	3 500.054 2	3 500.053 6	3 500.053 2	0.054 15	0.053 60	0.053 20
21	3.5	3 499.588 0	3 499.587 5	3 499.585 7	-0.412 00	-0.412 55	-0.414 35
22	4.0	3 999.259 1	3 999.257 9	3 999.260 0	-0.740 95	-0.742 15	-0.740 00
23	4.0	3 999.053 2	3 999.053 3	3 999.053 3	-0.946 85	-0.946 75	-0.946 75
24	4.0	4 000.384 1	4 000.384 4	4 000.383 9	0.384 10	0.384 40	0.383 85
25	4.5	4 499.975 1	4 499.974 9	4 499.975 4	-0.024 95	-0.025 15	-0.024 60
26	4.5	4 499.818 9	4 499.820 0	4 499.819 3	-0.181 15	-0.180 00	-0.180 75
27	4.5	4 499.713 9	4 499.715 1	4 499.714 7	-0.286 10	-0.284 95	-0.285 35
28	5.0	4 999.025 8	4 999.025 9	4 999.024 5	-0.974 25	-0.974 15	-0.975 50
29	5.0	4 999.148 4	4 999.148 9	4 999.149 1	-0.851 65	-0.851 15	-0.850 95
30	5.0	5 000.015 6	5 000.014 1	5 000.016 1	0.015 55	0.014 10	0.016 10
31	5.0	5 000.247 6	5 000.248 4	5 000.248 3	0.247 55	0.248 35	0.248 30

The mean values and standard deviations of the errors of indication of the three series are calculated as follows:

No.	Load	Errors	Mean Value	Standard Deviation
	g	g	mg	mg
1	5.0	e ₅	0.247 00	0.000 69
2	0.5	e _{0.5}	0.455 42	0.000 43
3	0.5	e _{0.5}	0.300 43	0.000 50
4	1.0	e _{1.0}	0.756 13	0.000 67
5	1.0	e _{1.0}	-0.233 88	0.000 29
6	1.0	e _{1.0}	-0.109 25	0.000 60
7	1.5	e _{1.5}	0.222 07	0.000 25
8	1.5	e _{1.5}	0.191 38	0.000 60
9	1.5	e _{1.5}	0.346 27	0.000 88
10	2.0	e _{2.0}	-0.601 85	0.000 53
11	2.0	e _{2.0}	-0.136 98	0.000 19
12	2.0	e _{2.0}	-0.343 60	0.000 43
13	2.5	e _{2.5}	-0.146 72	0.000 58
14	2.5	e _{2.5}	0.164 17	0.000 25
15	2.5	e _{2.5}	-0.042 83	0.001 33
16	3.0	e _{3.0}	-0.837 75	0.000 18
17	3.0	e _{3.0}	-0.247 28	0.000 12
18	3.0	e _{3.0}	0.153 03	0.000 46
19	3.5	e _{3.5}	-0.380 72	0.000 59
20	3.5	e _{3.5}	0.053 65	0.000 48
21	3.5	e _{3.5}	-0.412 97	0.001 23
22	4.0	e _{4.0}	-0.741 03	0.001 08
23	4.0	e _{4.0}	-0.946 78	0.000 06
24	4.0	e _{4.0}	0.384 12	0.000 28
25	4.5	e _{4.5}	-0.024 90	0.000 28
26	4.5	e _{4.5}	-0.180 63	0.000 58
27	4.5	e _{4.5}	-0.285 47	0.000 58
28	5.0	e _{5.0}	-0.974 63	0.000 75
29	5.0	e _{5.0}	-0.851 25	0.000 36
30	5.0	e _{5.0}	0.015 25	0.001 03
31	5.0	e _{5.0}	0.248 07	0.000 45

According to the mathematical matrix model of (9.1-5), the matrix *A* is made up from the measurement scheme and the combination of weights used:

No.	Test test	Errors of Indication / mg										Conventional mass correction of auxiliary weights / mg					
		$e_{0.5}$	$e_{1.0}$	$e_{1.5}$	$e_{2.0}$	$e_{2.5}$	$e_{3.0}$	$e_{3.5}$	$e_{4.0}$	$e_{4.5}$	$e_{5.0}$	$m_{0.5}$	$m_{0.5^*}$	m_1	m_{1^*}	m_2	m_{2^*}
1	5.0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0	0
2	0.5	1	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0
3	0.5	1	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0
4	1.0	0	1	0	0	0	0	0	0	0	0	1	1	0	0	0	0
5	1.0	0	1	0	0	0	0	0	0	0	0	0	0	1	0	0	0
6	1.0	0	1	0	0	0	0	0	0	0	0	0	0	0	1	0	0
7	1.5	0	0	1	0	0	0	0	0	0	0	1	0	1	0	0	0
8	1.5	0	0	1	0	0	0	0	0	0	0	0	1	0	1	0	0
9	1.5	0	0	1	0	0	0	0	0	0	0	1	0	0	1	0	0
10	2.0	0	0	0	1	0	0	0	0	0	0	0	0	0	0	1	0
11	2.0	0	0	0	1	0	0	0	0	0	0	0	0	0	0	0	1
12	2.0	0	0	0	1	0	0	0	0	0	0	0	0	1	1	0	0
13	2.5	0	0	0	0	1	0	0	0	0	0	1	0	0	0	1	0
14	2.5	0	0	0	0	1	0	0	0	0	0	0	1	0	0	0	1
15	2.5	0	0	0	0	1	0	0	0	0	0	0	1	1	1	0	0
16	3.0	0	0	0	0	0	1	0	0	0	0	0	0	1	0	1	0
17	3.0	0	0	0	0	0	1	0	0	0	0	0	0	0	1	0	1
18	3.0	0	0	0	0	0	1	0	0	0	0	1	1	0	0	1	0
19	3.5	0	0	0	0	0	0	1	0	0	0	1	0	1	0	1	0
20	3.5	0	0	0	0	0	0	1	0	0	0	0	1	0	1	0	1
21	3.5	0	0	0	0	0	0	1	0	0	0	0	1	0	1	1	0
22	4.0	0	0	0	0	0	0	0	1	0	0	0	0	0	0	1	1
23	4.0	0	0	0	0	0	0	0	1	0	0	0	0	1	1	1	0
24	4.0	0	0	0	0	0	0	0	1	0	0	1	1	1	0	0	1
25	4.5	0	0	0	0	0	0	0	0	1	0	1	0	1	1	0	1
26	4.5	0	0	0	0	0	0	0	0	1	0	0	1	1	1	0	1
27	4.5	0	0	0	0	0	0	0	0	1	0	1	0	0	0	1	1
28	5.0	0	0	0	0	0	0	0	0	0	1	0	0	1	0	1	1
29	5.0	0	0	0	0	0	0	0	0	0	1	0	0	0	1	1	1
30	5.0	0	0	0	0	0	0	0	0	0	1	1	1	0	0	1	1
31	5.0	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0	0

Therefore, the matrix $A_{31 \times 16}$ is equal to:

1	0	0	0	0	0	0	0	0	0	1	0	0	0	0	0
1	0	0	0	0	0	0	0	0	0	0	1	0	0	0	0
1	0	0	0	0	0	0	0	0	0	0	0	1	0	0	0
0	1	0	0	0	0	0	0	0	0	0	1	1	0	0	0
0	1	0	0	0	0	0	0	0	0	0	0	0	1	0	0
0	1	0	0	0	0	0	0	0	0	0	0	0	0	1	0
0	0	1	0	0	0	0	0	0	0	0	1	0	1	0	0
0	0	1	0	0	0	0	0	0	0	0	0	1	0	1	0
0	0	1	0	0	0	0	0	0	0	0	1	0	0	1	0
0	0	0	1	0	0	0	0	0	0	0	0	0	0	0	1
0	0	0	1	0	0	0	0	0	0	0	0	0	0	0	1
0	0	0	0	1	0	0	0	0	0	0	1	0	0	0	1
0	0	0	0	1	0	0	0	0	0	0	1	0	0	0	1
0	0	0	0	1	0	0	0	0	0	0	1	1	1	0	0
0	0	0	0	0	1	0	0	0	0	0	0	1	0	1	0
0	0	0	0	0	1	0	0	0	0	0	0	0	1	0	1
0	0	0	0	0	0	1	0	0	0	0	1	1	0	0	1
0	0	0	0	0	0	1	0	0	0	0	1	1	0	0	1
0	0	0	0	0	0	0	1	0	0	0	1	0	1	0	1
0	0	0	0	0	0	0	1	0	0	0	1	0	1	0	1
0	0	0	0	0	0	0	0	1	0	0	0	1	1	1	0
0	0	0	0	0	0	0	0	1	0	0	1	1	1	0	0
0	0	0	0	0	0	0	0	0	1	0	1	1	1	0	0
0	0	0	0	0	0	0	0	0	1	1	1	0	0	1	1
0	0	0	0	0	0	0	0	0	1	0	0	0	0	0	0

To make up the vector $Y = I - \delta m_R P - B$, it follows that:

$$\begin{matrix} I_{31 \times 1} = & \left| \begin{array}{c} 0.247\ 000 \\ 0.455\ 417 \\ 0.300\ 433 \\ 0.756\ 133 \\ -0.233\ 883 \\ -0.109\ 250 \\ 0.222\ 067 \\ 0.191\ 383 \\ 0.346\ 267 \\ -0.601\ 850 \\ -0.136\ 983 \\ -0.343\ 600 \\ -0.146\ 717 \\ 0.164\ 167 \\ -0.042\ 833 \\ -0.837\ 750 \\ -0.247\ 283 \\ 0.153\ 033 \\ -0.380\ 717 \\ 0.053\ 650 \\ -0.412\ 967 \\ -0.741\ 033 \\ -0.946\ 783 \\ 0.384\ 117 \\ -0.024\ 900 \\ -0.180\ 633 \\ -0.285\ 467 \\ -0.974\ 633 \\ -0.851\ 250 \\ 0.015\ 250 \\ 0.248\ 067 \end{array} \right. & P_{31 \times 1} = & \left| \begin{array}{c} 1 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 1 \end{array} \right. \end{matrix}$$

For the air buoyancy correction, the calculation of the air density with its uncertainty and the volumes of the reference weight and auxiliary weights with their uncertainties is required.

The global air density during calibration ρ_a , is calculated by averaging all values of temperature t , relative humidity hr , and barometric pressure p , from the three measurement series. These data are used as input quantities to calculate ρ_a (the output quantity) with the CIPM-2007 equation [9].

The standard uncertainties of the three input quantities u_t , u_{hr} and u_p , are obtained by considering the variability of the measurements, the uncertainty of the resolution and the uncertainty of the calibration of the measuring instruments. These uncertainties contribute to the calculation of the standard uncertainty of the air density u_{ρ_a} .

Based on the above and considering the information from measurement instruments of the environmental conditions, the best estimates of t , hr , p and ρ_a are obtained with their respective standard uncertainties u_t , u_{hr} , u_p and u_{ρ_a} :

Data of Environmental Conditions Measurement Instruments			
Instrument	Correction	$U, (k = 2)$	Resolution
Thermometer for t	0.00 °C	0.20 °C	0.1 °C
Hygrometer for hr	0.0 %	0.2 %	0.1 %
Barometer for p	0.0 hPa	0.2 hPa	0.1 hPa

Values of t, hr, p y ρ_a with their respective standard uncertainties			
Input quantity	Best estimate	Uncertainty, $k = 1$	
t	19.848 5 °C	u_t	0.165 9 °C
hr	52.157 6 %	u_{hr}	0.251 2 %
p	752.457 6 hPa	u_p	0.174 9 hPa

Output quantity	Best estimate	Uncertainty, $k = 1$	
ρ_a	0.000 889 49 g cm ⁻³	u_{ρ_a}	0.000 000 60 g cm ⁻³

Equation (9.1-3b) is used to calculate the air buoyancy corrections corresponding to the rows of the measurement scheme at the sampling point.

$$b_i = -(\rho_a - \rho_0)(V_i - m_N/\rho_c)$$

In other words, the correction for air buoyancy is calculated for each combination of auxiliary weights and the reference weight used in each measurement.

The values used to calculate each air buoyancy correction b_i are summarized below:

Volumes of auxiliary weights and reference weight								Densities	
Nominal value:	0.5 g	0.5 g	1 g	1 g	2 g	2 g	5 g	ρ_a in g cm ⁻³	0.000 889 49
Volume in cm ³ :	$V_{0.5}$	$V_{0.5*}$	V_1	V_{1*}	V_2	V_{2*}	V_R	ρ_0 in g cm ⁻³	0.001 2
	0.063 0	0.063 0	0.125 7	0.125 7	0.251 5	0.251 3	0.629 3	ρ_c in g cm ⁻³	8.0
u , Volume in cm ³ ($k = 1$)	$u_{V_{0.5}}$	$u_{V_{0.5*}}$	u_{V_1}	$u_{V_{1*}}$	u_{V_2}	$u_{V_{2*}}$	u_{V_R}	Air buoyancy correction	
	0.000 4	0.000 4	0.000 4	0.000 45	0.000 4	0.000 6	0.000 75	$b_i = -(\rho_a - \rho_0) \left(V_i - \frac{m_N}{\rho_c} \right)$	

With this information and following the measurement scheme, B is obtained as follows:

SIM Guidelines on the calibration of non-automatic weighing instruments with resolution less than 0.010 mg

ID m_N/g	$m_{0.5}$	$m_{0.5^*}$	m_1	m_{1^*}	m_2	m_{2^*}	m_R	b_i in g	b_i in mg
5.0	0	0	0	0	0	0	1	$1.335\ 2 \times 10^{-6}$	$1.335\ 2 \times 10^{-3}$
0.5	1	0	0	0	0	0	0	$1.552\ 6 \times 10^{-7}$	$1.552\ 6 \times 10^{-4}$
0.5	0	1	0	0	0	0	0	$1.552\ 6 \times 10^{-7}$	$1.552\ 6 \times 10^{-4}$
1.0	1	1	0	0	0	0	0	$3.105\ 1 \times 10^{-7}$	$3.105\ 1 \times 10^{-4}$
1.0	0	0	1	0	0	0	0	$2.173\ 6 \times 10^{-7}$	$2.173\ 6 \times 10^{-4}$
1.0	0	0	0	1	0	0	0	$2.173\ 6 \times 10^{-7}$	$2.173\ 6 \times 10^{-4}$
1.5	1	0	1	0	0	0	0	$3.726\ 2 \times 10^{-7}$	$3.726\ 2 \times 10^{-4}$
1.5	0	1	0	1	0	0	0	$3.726\ 2 \times 10^{-7}$	$3.726\ 2 \times 10^{-4}$
1.5	1	0	0	1	0	0	0	$3.726\ 2 \times 10^{-7}$	$3.726\ 2 \times 10^{-4}$
2.0	0	0	0	0	1	0	0	$4.657\ 7 \times 10^{-7}$	$4.657\ 7 \times 10^{-4}$
2.0	0	0	0	0	0	1	0	$4.036\ 7 \times 10^{-7}$	$4.036\ 7 \times 10^{-4}$
2.0	0	0	1	1	0	0	0	$4.347\ 2 \times 10^{-7}$	$4.347\ 2 \times 10^{-4}$
2.5	1	0	0	0	1	0	0	$6.210\ 3 \times 10^{-7}$	$6.210\ 3 \times 10^{-4}$
2.5	0	1	0	0	0	1	0	$5.589\ 3 \times 10^{-7}$	$5.589\ 3 \times 10^{-4}$
2.5	0	1	1	1	0	0	0	$5.899\ 8 \times 10^{-7}$	$5.899\ 8 \times 10^{-4}$
3.0	0	0	1	0	1	0	0	$6.831\ 3 \times 10^{-7}$	$6.831\ 3 \times 10^{-4}$
3.0	0	0	0	1	0	1	0	$6.210\ 3 \times 10^{-7}$	$6.210\ 3 \times 10^{-4}$
3.0	1	1	0	0	1	0	0	$7.762\ 9 \times 10^{-7}$	$7.762\ 9 \times 10^{-4}$
3.5	1	0	1	0	1	0	0	$8.383\ 9 \times 10^{-7}$	$8.383\ 9 \times 10^{-4}$
3.5	0	1	0	1	0	1	0	$7.762\ 9 \times 10^{-7}$	$7.762\ 9 \times 10^{-4}$
3.5	0	1	0	1	1	0	0	$8.383\ 9 \times 10^{-7}$	$8.383\ 9 \times 10^{-4}$
4.0	0	0	0	0	1	1	0	$8.694\ 4 \times 10^{-7}$	$8.694\ 4 \times 10^{-4}$
4.0	0	0	1	1	1	0	0	$9.004\ 9 \times 10^{-7}$	$9.004\ 9 \times 10^{-4}$
4.0	1	1	1	0	0	1	0	$9.315\ 4 \times 10^{-7}$	$9.315\ 4 \times 10^{-4}$
4.5	1	0	1	1	0	1	0	$9.936\ 5 \times 10^{-7}$	$9.936\ 5 \times 10^{-4}$
4.5	0	1	1	1	0	1	0	$9.936\ 5 \times 10^{-7}$	$9.936\ 5 \times 10^{-4}$
4.5	1	0	0	0	1	1	0	$1.024\ 7 \times 10^{-6}$	$1.024\ 7 \times 10^{-3}$
5.0	0	0	1	0	1	1	0	$1.086\ 8 \times 10^{-6}$	$1.086\ 8 \times 10^{-3}$
5.0	0	0	0	1	1	1	0	$1.086\ 8 \times 10^{-6}$	$1.086\ 8 \times 10^{-3}$
5.0	1	1	0	0	1	1	0	$1.180\ 0 \times 10^{-6}$	$1.180\ 0 \times 10^{-3}$
5.0	0	0	0	0	0	0	1	$1.335\ 2 \times 10^{-6}$	$1.335\ 2 \times 10^{-3}$

Therefore, the vector B in milligrams is:

$$B_{31 \times 1} = \begin{pmatrix} 1.335\ 2 \times 10^{-3} \\ 1.552\ 6 \times 10^{-4} \\ 1.552\ 6 \times 10^{-4} \\ 3.105\ 1 \times 10^{-4} \\ 2.173\ 6 \times 10^{-4} \\ 2.173\ 6 \times 10^{-4} \\ 3.726\ 2 \times 10^{-4} \\ 3.726\ 2 \times 10^{-4} \\ 3.726\ 2 \times 10^{-4} \\ 4.657\ 7 \times 10^{-4} \\ 4.036\ 7 \times 10^{-4} \\ 4.347\ 2 \times 10^{-4} \\ 6.210\ 3 \times 10^{-4} \\ 5.589\ 3 \times 10^{-4} \\ 5.899\ 8 \times 10^{-4} \\ 6.831\ 3 \times 10^{-4} \\ 6.210\ 3 \times 10^{-4} \\ 7.762\ 9 \times 10^{-4} \\ 8.383\ 9 \times 10^{-4} \\ 7.762\ 9 \times 10^{-4} \\ 8.383\ 9 \times 10^{-4} \\ 8.694\ 4 \times 10^{-4} \\ 9.004\ 9 \times 10^{-4} \\ 9.315\ 4 \times 10^{-4} \\ 9.936\ 5 \times 10^{-4} \\ 9.936\ 5 \times 10^{-4} \\ 1.024\ 7 \times 10^{-3} \\ 1.086\ 8 \times 10^{-3} \\ 1.086\ 8 \times 10^{-3} \\ 1.180\ 0 \times 10^{-3} \\ 1.335\ 2 \times 10^{-3} \end{pmatrix}$$

Thus, the vector Y is:

I mg	$-\delta m_R$ mg	P	$-$	B mg	$=$	Y mg
0.247 000		1		$1.335\ 2 \times 10^{-3}$		0.001 665
0.455 417		0		$1.552\ 6 \times 10^{-4}$		0.455 261
0.300 433		0		$1.552\ 6 \times 10^{-4}$		0.300 278
0.756 133		0		$3.105\ 1 \times 10^{-4}$		0.755 823
-0.233 883		0		$2.173\ 6 \times 10^{-4}$		-0.234 101
-0.109 250		0		$2.173\ 6 \times 10^{-4}$		-0.109 467
0.222 067		0		$3.726\ 2 \times 10^{-4}$		0.221 694
0.191 383		0		$3.726\ 2 \times 10^{-4}$		0.191 011
0.346 267		0		$3.726\ 2 \times 10^{-4}$		0.345 894
-0.601 850		0		$4.657\ 7 \times 10^{-4}$		-0.602 316
-0.136 983		0		$4.036\ 7 \times 10^{-4}$		-0.137 387
-0.343 600		0		$4.347\ 2 \times 10^{-4}$		-0.344 035
-0.146 717		0		$6.210\ 3 \times 10^{-4}$		-0.147 338
0.164 167		0		$5.589\ 3 \times 10^{-4}$		0.163 608
-0.042 833		0		$5.899\ 8 \times 10^{-4}$		-0.043 423
-0.837 750	-(0.244)	0	-	$6.831\ 3 \times 10^{-4}$	=	-0.838 433
-0.247 283		0		$6.210\ 3 \times 10^{-4}$		-0.247 904
0.153 033		0		$7.762\ 9 \times 10^{-4}$		0.152 257
-0.380 717		0		$8.383\ 9 \times 10^{-4}$		-0.381 555
0.053 650		0		$7.762\ 9 \times 10^{-4}$		0.052 874
-0.412 967		0		$8.383\ 9 \times 10^{-4}$		-0.413 805
-0.741 033		0		$8.694\ 4 \times 10^{-4}$		-0.741 903
-0.946 783		0		$9.004\ 9 \times 10^{-4}$		-0.947 684
0.384 117		0		$9.315\ 4 \times 10^{-4}$		0.383 185
-0.024 900		0		$9.936\ 5 \times 10^{-4}$		-0.025 894
-0.180 633		0		$9.936\ 5 \times 10^{-4}$		-0.181 627
-0.285 467		0		$1.024\ 7 \times 10^{-3}$		-0.286 491
-0.974 633		0		$1.086\ 8 \times 10^{-3}$		-0.975 720
-0.851 250		0		$1.086\ 8 \times 10^{-3}$		-0.852 337
0.015 250		0		$1.180\ 0 \times 10^{-3}$		0.014 070
0.248 067		1		$1.335\ 2 \times 10^{-3}$		0.002 731

Therefore, the vector of estimates E is calculated with equation (9.1-6):

$$(A_{16 \times 31}^T A_{31 \times 16})_{16 \times 16}^{-1} A_{16 \times 31}^T Y_{31 \times 1} = E_{16 \times 1}$$

		E	E
		in mg	in μg
$E =$	$e_{0.5}$	0.000 148	0.148 2
	$e_{1.0}$	0.000 752	0.752 3
	$e_{1.5}$	0.001 306	1.305 6
	$e_{2.0}$	0.001 678	1.678 0
	$e_{2.5}$	0.002 124	2.124 1
	$e_{3.0}$	0.001 373	1.373 2
	$e_{3.5}$	0.002 089	2.089 0
	$e_{4.0}$	0.001 781	1.780 7
	$e_{4.5}$	0.002 410	2.410 3
	$e_{5.0}$	0.002 198	2.198 1
	$m_{0.5}$	0.455 240	455.240 3
	$m_{0.5^*}$	0.300 003	300.002 8
	m_1	-0.234 689	-234.688 9
	m_{1^*}	-0.110 556	-110.556 3
	m_2	-0.604 672	-604.671 5
	m_{2^*}	-0.138 855	-138.854 9

The first 10 elements of the vector E correspond to the errors of indication of the microbalance test points, and the last six elements are the conventional mass corrections of the auxiliary weights.

13.1.1.4 Uncertainty calculation

13.1.1.4.1 Component due to the residuals of the least squares adjustment,

$$u_{resid}^2$$

To obtain the value of u_{resid}^2 it is necessary to calculate the vector of residuals R with equation (9.1-5). From (10.3-1) we obtain u_{resid}^2 as follows:

Y mg	–	AE mg	=	R mg
0.001 665		0.002 198		-0.000 533
0.455 261		0.455 388		-0.000 127
0.300 278		0.300 151		0.000 127
0.755 823		0.755 995		-0.000 173
-0.234 101		-0.233 937		-0.000 164
-0.109 467		-0.109 804		0.000 337
0.221 694		0.221 857		-0.000 163
0.191 011		0.190 752		0.000 259
0.345 894		0.345 990		-0.000 096
-0.602 316		-0.602 994		0.000 678
-0.137 387		-0.137 177		-0.000 210
-0.344 035		-0.343 567		-0.000 468
-0.147 338		-0.147 307		-0.000 031
0.163 608		0.163 272		0.000 336
-0.043 423		-0.043 118		-0.000 305
-0.838 433	–	-0.837 987	=	-0.000 446
-0.247 904		-0.248 038		0.000 134
0.152 257		0.151 945		0.000 312
-0.381 555		-0.382 031		0.000 476
0.052 874		0.052 681		0.000 193
-0.413 805		-0.413 136		-0.000 669
-0.741 903		-0.741 746		-0.000 157
-0.947 684		-0.948 136		0.000 452
0.383 185		0.383 480		-0.000 295
-0.025 894		-0.026 449		0.000 556
-0.181 627		-0.181 687		0.000 060
-0.286 491		-0.285 876		-0.000 616
-0.975 720		-0.976 017		0.000 297
-0.852 337		-0.851 885		-0.000 452
0.014 070		0.013 915		0.000 155
0.002 731		0.002 198		0.000 533

$$u_{resid}^2 = \frac{(Y - AE)^T(Y - AE)}{m - n}$$

$$u_{resid}^2 = \frac{4.152\ 7 \times 10^{-6} \text{ mg}^2}{31 - 16}$$

$$u_{resid}^2 = 2.768\ 5 \times 10^{-7} \text{ mg}^2$$

$$u_{resid} = \sqrt{2.7685 \times 10^{-7} \text{ mg}^2}$$

$$u_{resid} = 5.261\ 6 \times 10^{-4} \text{ mg}$$

$$u_{resid} = 0.526\ 16 \text{ }\mu\text{g}$$

13.1.1.4.2 Component due to repeatability of measurements, $u_{rep_i}^2$

The standard uncertainty due to the repeatability of the three measurement series u_{rep_i} , is calculated according to 10.1 as follows:

<i>i</i>	Load	Standard Deviation, S_i	$u_{rep_i} = \frac{S_i}{\sqrt{N}} = \frac{S_i}{\sqrt{3}}$	u_{rep_i}	$u_{rep_i}^2$	$u_{rep_i}^2$
	g	mg	mg	mg	mg ²	mg ²
1	5.0	0.000 69	0.000 401	0.401 0	$1.608 3 \times 10^{-7}$	0.160 8
2	0.5	0.000 43	0.000 246	0.245 5	$6.027 8 \times 10^{-8}$	0.060 3
3	0.5	0.000 50	0.000 291	0.290 6	$8.444 4 \times 10^{-8}$	0.084 4
4	1.0	0.000 67	0.000 388	0.387 7	$1.502 8 \times 10^{-7}$	0.150 3
5	1.0	0.000 29	0.000 169	0.169 1	$2.861 1 \times 10^{-8}$	0.028 6
6	1.0	0.000 60	0.000 346	0.346 4	$1.200 0 \times 10^{-7}$	0.120 0
7	1.5	0.000 25	0.000 142	0.142 4	$2.027 8 \times 10^{-8}$	0.020 3
8	1.5	0.000 60	0.000 349	0.349 2	$1.219 4 \times 10^{-7}$	0.121 9
9	1.5	0.000 88	0.000 509	0.508 5	$2.586 1 \times 10^{-7}$	0.258 6
10	2.0	0.000 53	0.000 304	0.304 1	$9.250 0 \times 10^{-8}$	0.092 5
11	2.0	0.000 19	0.000 109	0.109 3	$1.194 4 \times 10^{-8}$	0.011 9
12	2.0	0.000 43	0.000 250	0.250 0	$6.250 0 \times 10^{-8}$	0.062 5
13	2.5	0.000 58	0.000 335	0.334 6	$1.119 4 \times 10^{-7}$	0.111 9
14	2.5	0.000 25	0.000 142	0.142 4	$2.027 8 \times 10^{-8}$	0.020 3
15	2.5	0.001 33	0.000 769	0.768 8	$5.911 1 \times 10^{-7}$	0.591 1
16	3.0	0.000 18	0.000 104	0.104 1	$1.083 3 \times 10^{-8}$	0.010 8
17	3.0	0.000 12	0.000 067	0.066 7	$4.444 4 \times 10^{-9}$	0.004 4
18	3.0	0.000 46	0.000 268	0.268 2	$7.194 4 \times 10^{-8}$	0.071 9
19	3.5	0.000 59	0.000 338	0.338 3	$1.144 4 \times 10^{-7}$	0.114 4
20	3.5	0.000 48	0.000 275	0.275 4	$7.583 3 \times 10^{-8}$	0.075 8
21	3.5	0.001 23	0.000 710	0.709 7	$5.036 1 \times 10^{-7}$	0.503 6
22	4.0	0.001 08	0.000 622	0.622 0	$3.869 4 \times 10^{-7}$	0.386 9
23	4.0	0.000 06	0.000 033	0.033 3	$1.111 1 \times 10^{-9}$	0.001 1
24	4.0	0.000 28	0.000 159	0.159 0	$2.527 8 \times 10^{-8}$	0.025 3
25	4.5	0.000 28	0.000 161	0.160 7	$2.583 3 \times 10^{-8}$	0.025 8
26	4.5	0.000 58	0.000 337	0.337 1	$1.136 1 \times 10^{-7}$	0.113 6
27	4.5	0.000 58	0.000 337	0.337 1	$1.136 1 \times 10^{-7}$	0.113 6
28	5.0	0.000 75	0.000 434	0.434 3	$1.886 1 \times 10^{-7}$	0.188 6
29	5.0	0.000 36	0.000 208	0.208 2	$4.333 3 \times 10^{-8}$	0.043 3
30	5.0	0.001 03	0.000 597	0.596 5	$3.558 3 \times 10^{-7}$	0.355 8
31	5.0	0.000 45	0.000 259	0.258 7	$6.694 4 \times 10^{-8}$	0.066 9

13.1.1.4.3 Component due to resolution, u_{res}^2

The variance u_{res}^2 is obtained with equation (10.2-1):

$$u_{res}^2 = \left(\frac{d}{2\sqrt{3}} \sqrt{2} \right)^2 = \left(\frac{0.0001 \text{ mg}}{2\sqrt{3}} \sqrt{2} \right)^2 = 1.6667 \times 10^{-9} \text{ mg}^2$$

$$u_{res} = \sqrt{1.6667 \times 10^{-9} \text{ mg}^2} = 4.0825 \times 10^{-5} \text{ mg} = 0.0408 \mu\text{g}$$

13.1.1.4.4 Component due to air buoyancy correction, u_{bi}^2

The calculation of this contribution for the scheme that was used in the combination of the weights used for each row of the measurement series is done with equation (10.4-1b).

The data required to calculate the contribution due to air buoyancy correction are summarized below:

Volumes of auxiliary weights and reference weights								Densities	
Nominal value:	0.5 g	0.5 g	1 g	1 g	2 g	2 g	5 g	u_{ρ_a} in g cm ⁻³	0.000 000 60
Volume in cm ³ :	$V_{0.5}$	$V_{0.5^*}$	V_1	V_{1^*}	V_2	V_{2^*}	V_R	ρ_0 in g cm ⁻³	0.001 2
	0.063 0	0.063 0	0.125 7	0.125 7	0.251 5	0.251 3	0.629 3	ρ_c in g cm ⁻³	8.0
u , Volume in cm ³ ($k = 1$)	$u_{V_{0.5}}$	$u_{V_{0.5^*}}$	u_{V_1}	$u_{V_{1^*}}$	u_{V_2}	$u_{V_{2^*}}$	u_{V_R}	Uncertainty due to air buoyancy	
	0.000 4	0.000 4	0.000 4	0.000 45	0.000 4	0.000 6	0.000 75	$u_{bi} = \sqrt{\left(V_i - \frac{m_{Ni}}{\rho_c} \right)^2 u_{\rho_a}^2 + (\rho_a - \rho_0)^2 u_{V_i}^2}$	

SIM Guidelines on the calibration of non-automatic weighing instruments with resolution less than 0.010 mg

ID m_N / g	$m_{0.5}$	$m_{0.5^*}$	m_1	m_{1^*}	m_2	m_{2^*}	m_R	$\left(V_i - \frac{m_{N_i}}{\rho_c}\right)^2 u^2(\rho_a)$ in mg^2	$(\rho_a - \rho_0)^2 u^2(V_i)$ in mg^2	$u_{b_i}^2$ in mg^2
5.0	0	0	0	0	0	0	1	$6.625 5 \times 10^{-18}$	$5,423 6 \times 10^{-14}$	$5,424 2 \times 10^{-14}$
0.5	1	0	0	0	0	0	0	$8.958 2 \times 10^{-20}$	$1,542 7 \times 10^{-14}$	$1,542 7 \times 10^{-14}$
0.5	0	1	0	0	0	0	0	$8.958 2 \times 10^{-20}$	$1,542 7 \times 10^{-14}$	$1,542 7 \times 10^{-14}$
1.0	1	1	0	0	0	0	0	$3.583 3 \times 10^{-19}$	$3.085 4 \times 10^{-14}$	$3.085 5 \times 10^{-14}$
1.0	0	0	1	0	0	0	0	$1.755 8 \times 10^{-19}$	$1,542 7 \times 10^{-14}$	$1,542 7 \times 10^{-14}$
1.0	0	0	0	1	0	0	0	$1.755 8 \times 10^{-19}$	$1,952 5 \times 10^{-14}$	$1,952 5 \times 10^{-14}$
1.5	1	0	1	0	0	0	0	$5.159 9 \times 10^{-19}$	$3.085 4 \times 10^{-14}$	$3.085 5 \times 10^{-14}$
1.5	0	1	0	1	0	0	0	$5.159 9 \times 10^{-19}$	$3.495 2 \times 10^{-14}$	$3.495 2 \times 10^{-14}$
1.5	1	0	0	1	0	0	0	$5.159 9 \times 10^{-19}$	$3,495 2 \times 10^{-14}$	$3,495 2 \times 10^{-14}$
2.0	0	0	0	0	1	0	0	$8.062 4 \times 10^{-19}$	$1,542 7 \times 10^{-14}$	$1,542 8 \times 10^{-14}$
2.0	0	0	0	0	0	1	0	$6.055 8 \times 10^{-19}$	$3,471 1 \times 10^{-14}$	$3,471 2 \times 10^{-14}$
2.0	0	0	1	1	0	0	0	$7.023 2 \times 10^{-19}$	$3,495 2 \times 10^{-14}$	$3,495 3 \times 10^{-14}$
2.5	1	0	0	0	1	0	0	$1.433 3 \times 10^{-18}$	$3.085 4 \times 10^{-14}$	$3.085 6 \times 10^{-14}$
2.5	0	1	0	0	0	1	0	$1.161 0 \times 10^{-18}$	$5.013 8 \times 10^{-14}$	$5.013 9 \times 10^{-14}$
2.5	0	1	1	1	0	0	0	$1,293 6 \times 10^{-18}$	$5.037 9 \times 10^{-14}$	$5.038 0 \times 10^{-14}$
3.0	0	0	1	0	1	0	0	$1,734 3 \times 10^{-18}$	$3.085 4 \times 10^{-14}$	$3.085 6 \times 10^{-14}$
3.0	0	0	0	1	0	1	0	$1.433 3 \times 10^{-18}$	$5,423 6 \times 10^{-14}$	$5,423 7 \times 10^{-14}$
3.0	1	1	0	0	1	0	0	$2,239 6 \times 10^{-18}$	$4.628 1 \times 10^{-14}$	$4,628 3 \times 10^{-14}$
3.5	1	0	1	0	1	0	0	$2,612 2 \times 10^{-18}$	$4.628 1 \times 10^{-14}$	$4,628 4 \times 10^{-14}$
3.5	0	1	0	1	0	1	0	$2,239 6 \times 10^{-18}$	$6,966 3 \times 10^{-14}$	$6,966 5 \times 10^{-14}$
3.5	0	1	0	1	1	0	0	$2,612 2 \times 10^{-18}$	$5.037 9 \times 10^{-14}$	$5.038 2 \times 10^{-14}$
4.0	0	0	0	0	1	1	0	$2,809 3 \times 10^{-18}$	$5.013 8 \times 10^{-14}$	$5.014 1 \times 10^{-14}$
4.0	0	0	1	1	1	0	0	$3.013 5 \times 10^{-18}$	$5.037 9 \times 10^{-14}$	$5.038 2 \times 10^{-14}$
4.0	1	1	1	0	0	1	0	$3.225 0 \times 10^{-18}$	$8,099 2 \times 10^{-14}$	$8.099 5 \times 10^{-14}$
4.5	1	0	1	1	0	1	0	$3.669 3 \times 10^{-18}$	$8.509 0 \times 10^{-14}$	$8,509 4 \times 10^{-14}$
4.5	0	1	1	1	0	1	0	$3.669 3 \times 10^{-18}$	$8.509 0 \times 10^{-14}$	$8,509 4 \times 10^{-14}$
4.5	1	0	0	0	1	1	0	$3.902 2 \times 10^{-18}$	$6.556 5 \times 10^{-14}$	$6,556 9 \times 10^{-14}$
5.0	0	0	1	0	1	1	0	$4.389 5 \times 10^{-18}$	$6.556 5 \times 10^{-14}$	$6,556 9 \times 10^{-14}$
5.0	0	0	0	1	1	1	0	$4.389 5 \times 10^{-18}$	$6,966 3 \times 10^{-14}$	$6,966 7 \times 10^{-14}$
5.0	1	1	0	0	1	1	0	$5.174 3 \times 10^{-18}$	$8,099 2 \times 10^{-14}$	$8,099 7 \times 10^{-14}$
5.0	0	0	0	0	0	0	1	$6.625 5 \times 10^{-18}$	$5,423 6 \times 10^{-14}$	$5,424 2 \times 10^{-14}$

13.1.1.4.5 Component due to the correction of the conventional mass of the air reference weight, $u_{m_R}^2$

According to equation (10.5-1), this contribution is obtained from the calibration certificate u_{cal} and the instability of the mass value u_{inst} of the reference weight. For the example shown it is given that:

$$u_{m_R}^2 = u_{cal}^2 + u_{inst}^2 = (0.0025 \text{ mg})^2 + (0 \text{ mg})^2 = 6.25 \times 10^{-6} \text{ mg}^2 = 6.25 \mu\text{g}^2$$

13.1.1.4.6 Construction of the U_Y matrix

As explained in Section 10, the covariance matrix U_Y is constructed with the covariance matrices of each uncertainty component: u_{res}^2 , u_{resid}^2 , $u_{rep_i}^2$, $u_{b_i}^2$ and $u_{m_R}^2$.

Considering 10.2 and 10.3, the covariance matrices for the components due to repeatability U_{res} and the residuals from the least squares fitting U_{resid} are:

$$U_{res} = u_{res}^2 I_{d_{31 \times 31}}$$

$$U_{resid} = u_{resid}^2 I_{d_{31 \times 31}}$$

Where $I_{d_{31 \times 31}}$ is an identity matrix with dimension 31×31 .

This results in:

$$U_{res_{31 \times 31}} = \begin{vmatrix} u_{res}^2 & 0 & \dots & 0 & 0 \\ 0 & u_{res}^2 & \dots & 0 & 0 \\ \vdots & 0 & \ddots & 0 & \vdots \\ 0 & 0 & \dots & u_{res}^2 & 0 \\ 0 & 0 & \dots & 0 & u_{res}^2 \end{vmatrix}$$

$$U_{res_{31 \times 31}} \text{ in } \text{mg}^2 = \begin{vmatrix} 1,6667 \times 10^{-9} & 0 & \dots & 0 & 0 \\ 0 & 1,6667 \times 10^{-9} & \dots & 0 & 0 \\ \vdots & 0 & \ddots & 0 & \vdots \\ 0 & 0 & \dots & 1,6667 \times 10^{-9} & 0 \\ 0 & 0 & \dots & 0 & 1,6667 \times 10^{-9} \end{vmatrix}$$

$$\mathbf{U}_{\text{resid}}_{31 \times 31} = \begin{vmatrix} u_{\text{resid}}^2 & 0 & \dots & 0 & 0 \\ 0 & u_{\text{resid}}^2 & \dots & 0 & 0 \\ \vdots & 0 & \ddots & 0 & \vdots \\ 0 & 0 & \dots & u_{\text{resid}}^2 & 0 \\ 0 & 0 & \dots & 0 & u_{\text{resid}}^2 \end{vmatrix}$$

$$\mathbf{U}_{\text{resid}}_{31 \times 31} \text{ in mg}^2 = \begin{vmatrix} 2,768\ 5 \times 10^{-7} & 0 & \dots & 0 & 0 \\ 0 & 2,768\ 5 \times 10^{-7} & \dots & 0 & 0 \\ \vdots & 0 & \ddots & 0 & \vdots \\ 0 & 0 & \dots & 2,768\ 5 \times 10^{-7} & 0 \\ 0 & 0 & \dots & 0 & 2,768\ 5 \times 10^{-7} \end{vmatrix}$$

From 10.1 and 10.4, the covariance matrices for the components due to repeatability and air buoyancy correction are constructed by generating a square matrix with dimension 31×31 whose diagonal elements are the variances $u_{\text{rep}_i}^2$ for \mathbf{U}_{rep} and $u_{b_i}^2$ for \mathbf{U}_b respectively; values outside the diagonal are equal to zero, i.e:

$$(\mathbf{U}_{\text{rep}})_{ij} = \begin{cases} u_{\text{rep}_i}^2 & \text{para } i = j \\ 0 & \text{para } i \neq j \end{cases}$$

$$(\mathbf{U}_b)_{ij} = \begin{cases} u_{b_i}^2 & \text{para } i = j \\ 0 & \text{para } i \neq j \end{cases}$$

The variance matrix of the component variance due to the conventional mass correction of the reference weight U_{m_R} is constructed according to 10.5 as follows:

$$U_{m_R} = u_{m_R}^2 PP^T$$

For the example, U_{m_R} is a 31×31 square matrix where the corner elements of the matrix contain the value $u_{m_R}^2$; the other elements are zero:

$$(U_{m_R})_{1,1} = (U_{m_R})_{31,1} = (U_{m_R})_{1,31} = (U_{m_R})_{31,31} = u_{m_R}^2 = 6.25 \times 10^{-6} \text{ mg}^2$$

$$U_{m_R 31 \times 31} = \begin{vmatrix} u_{m_R}^2 & 0 & \dots & 0 & u_{m_R}^2 \\ 0 & 0 & \dots & 0 & 0 \\ \vdots & 0 & \ddots & 0 & \vdots \\ 0 & 0 & \dots & 0 & 0 \\ u_{m_R}^2 & 0 & \dots & 0 & u_{m_R}^2 \end{vmatrix}$$

$$U_{m_R 31 \times 31} \text{ in mg}^2 = \begin{vmatrix} 6.25 \times 10^{-6} & 0 & \dots & 0 & 6.25 \times 10^{-6} \\ 0 & 0 & \dots & 0 & 0 \\ \vdots & 0 & \ddots & 0 & \vdots \\ 0 & 0 & \dots & 0 & 0 \\ 6.25 \times 10^{-6} & 0 & \dots & 0 & 6.25 \times 10^{-6} \end{vmatrix}$$

Having the covariance matrices of each of the sources of uncertainty, U_Y is obtained from equation (10-3):

$$U_Y = U_{\text{res}} + U_{\text{resid}} + U_{\text{rep}} + U_{\text{b}} + U_{m_R}$$

whose dimension is 31×31 for the case of the example.

13.1.1.4.7 Calculation of the covariance matrix of the estimates, U_E

For the example, U_E is obtained with equation (10-1), therefore:

$$U_{E_{16 \times 16}} = (A_{16 \times 31}^T A_{31 \times 16})_{16 \times 16}^{-1} A_{16 \times 31}^T U_{Y_{31 \times 31}} A_{31 \times 16} (A_{16 \times 31}^T A_{31 \times 16})_{16 \times 16}^{-1}$$

U_E matrix (Values $\times 10^{-6} \text{ mg}^2$)

0.285 30	0.166 85	0.257 72	0.269 45	0.37153	0.428 18	0.515 44	0.573 03	0.648 62	0.664 24	-0.113 71	-0.105 80	-0.138 68	-0.152 54	-0.263 28	-0.254 95	0.285 30
0.166 85	0.434 66	0.466 58	0.550 04	0.69934	0.836 99	0.992 19	1.127 19	1.268 89	1.328 48	-0.170 96	-0.162 28	-0.297 18	-0.301 31	-0.529 89	-0.523 42	0.166 85
0.257 72	0.466 58	0.850 63	0.823 34	1.05008	1.256 73	1.491 84	1.688 02	1.913 89	1.992 72	-0.278 48	-0.235 21	-0.433 62	-0.465 52	-0.789 91	-0.783 28	0.257 72
0.269 45	0.550 04	0.823 34	1.203 45	1.35451	1.632 95	1.905 83	2.182 81	2.457 02	2.656 96	-0.271 09	-0.267 65	-0.556 56	-0.551 96	-1.081 05	-1.086 98	0.269 45
0.371 53	0.699 34	1.050 08	1.354 51	1.887 58	2.052 37	2.403 42	2.744 33	3.079 78	3.321 20	-0.365 99	-0.377 50	-0.680 13	-0.680 83	-1.353 75	-1.350 87	0.371 53
0.428 18	0.836 99	1.256 73	1.632 95	2.052 37	2.565 40	2.879 95	3.284 64	3.688 56	3.985 44	-0.426 81	-0.429 52	-0.826 46	-0.831 67	-1.633 72	-1.610 39	0.428 18
0.515 44	0.992 19	1.491 84	1.905 83	2.403 42	2.879 95	3.542 94	3.845 39	4.325 49	4.649 68	-0.514 40	-0.516 81	-0.969 20	-0.985 07	-1.892 47	-1.875 92	0.515 44
0.573 03	1.127 19	1.688 02	2.182 81	2.744 33	3.284 64	3.845 39	4.527 99	4.939 59	5.313 92	-0.574 69	-0.571 32	-1.129 44	-1.104 30	-2.159 26	-2.156 11	0.573 03
0.648 62	1.268 89	1.913 89	2.457 02	3.079 78	3.688 56	4.325 49	4.939 59	5.709 73	5.978 16	-0.676 37	-0.619 11	-1.254 85	-1.260 58	-2.401 75	-2.450 03	0.648 62
0.664 24	1.328 48	1.992 72	2.656 96	3.321 20	3.985 44	4.649 68	5.313 92	5.978 16	6.642 40	-0.664 24	-0.664 24	-1.328 48	-1.328 48	-2.656 96	-2.656 96	0.664 24
-0.113 71	-0.170 96	-0.278 48	-0.271 09	-0.365 99	-0.426 81	-0.514 40	-0.574 69	-0.676 37	-0.664 24	0.155 61	0.069 13	0.133 87	0.167 12	0.249 21	0.262 47	-0.113 71
-0.105 80	-0.162 28	-0.235 21	-0.267 65	-0.377 50	-0.429 52	-0.516 81	-0.571 32	-0.619 11	-0.664 24	0.069 13	0.144 70	0.143 76	0.136 76	0.278 19	0.247 05	-0.105 80
-0.138 68	-0.297 18	-0.433 62	-0.556 56	-0.680 13	-0.826 46	-0.969 20	-1.129 44	-1.254 85	-1.328 48	0.133 87	0.143 76	0.335 94	0.266 61	0.529 10	0.538 40	-0.138 68
-0.152 54	-0.301 31	-0.465 52	-0.551 96	-0.680 83	-0.831 67	-0.985 07	-1.104 30	-1.260 58	-1.328 48	0.167 12	0.136 76	0.266 61	0.342 18	0.531 90	0.517 71	-0.152 54
-0.263 28	-0.529 89	-0.789 91	-1.081 05	-1.353 75	-1.633 72	-1.892 47	-2.159 26	-2.401 75	-2.656 96	0.249 21	0.278 19	0.529 10	0.531 90	1.126 93	1.062 11	-0.263 28
-0.254 95	-0.523 42	-0.783 28	-1.086 98	-1.350 87	-1.610 39	-1.875 92	-2.156 11	-2.450 03	-2.656 96	0.262 47	0.247 05	0.538 40	0.517 71	1.062 11	1.134 17	-0.254 95
0.285 30	0.166 85	0.257 72	0.269 45	0.371 53	0.428 18	0.515 44	0.573 03	0.648 62	0.664 24	-0.113 71	-0.105 80	-0.138 68	-0.152 54	-0.263 28	-0.254 95	0.285 30

13.1.1.4.8 Calculation of uncertainty of the estimates, E

The standard uncertainty u_{E_i} of each of the elements of the vector of estimates E , is obtained with the square root to the elements of the diagonal of the covariance matrix U_E , that is:

$$u_{E_i} = \sqrt{(U_E)_{ij}} \text{ para } i = j$$

The variability of the estimates is assumed to approximate a normal probability distribution. Therefore, the expanded uncertainty U_{E_i} for a confidence level of approximately 95 % is:

$$U_{E_i} = k u_{E_i} \text{ con } k = 2$$

13.1.1.4.9 Calibration results.

The following table shows the results of the calibration of the instrument indication errors and the conventional mass corrections of the auxiliary weights, both expressed in milligrams and micrograms.

Errors of Indication of the Instrument					
Test point	e_i mg	$U_{e_i}, (k = 2)$ mg		e_i μg	$U_{e_i}, (k = 2)$ μg
0.5 g	0.000 1	0.001 1		0.1	1.1
1.0 g	0.000 8	0.001 3		0.8	1.3
1.5 g	0.001 3	0.001 8		1.3	1.8
2.0 g	0.001 7	0.002 2		1.7	2.2
2.5 g	0.002 1	0.002 7		2.1	2.7
3.0 g	0.001 4	0.003 2		1.4	3.2
3.5 g	0.002 1	0.003 8		2.1	3.8
4.0 g	0.001 8	0.004 3		1.8	4.3
4.5 g	0.002 4	0.004 8		2.4	4.8
5.0 g	0.002 2	0.005 2		2.2	5.2

Conventional mass of auxiliary weights					
Nominal value	δm_i mg	$U_{\delta m_i}, (k = 2)$ mg		δm_i μg	$U_{\delta m_i}, (k = 2)$ μg
0.5 g	0.455 24	0.000 79		455.24	0.79
0.5 g	0.300 00	0.000 76		300.00	0.76
1 g	-0.234 7	0.00 12		-234.7	1.2
1 g	-0.110 6	0.00 12		-110.6	1.2
2 g	-0.604 7	0.00 21		-604.7	2.1
2 g	-0.138 9	0.00 21		-138.9	2.1

13.1.2 Appendix A 2: MATRIX MODELS

DATA RECORDING FORM *Matrix model for instruments with a maximum capacity of 5 g.*

		Planilla de toma de datos							1		
		Pesas							Indicación de la balanza		
carga/g		0,5	0,5*	1	1*	2	2*	5	Cero	Carga	Cero
		Temp/°C=		h relat./%=		Presión. atm./hPa=			Ajustar sensibilidad		
1	5	0	0	0	0	0	0	1			
2	0,5	1	0	0	0	0	0	0			
3	0,5	0	1	0	0	0	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=			Ajustar sensibilidad		
4	1	1	1	0	0	0	0	0			
5	1	0	0	1	0	0	0	0			
6	1	0	0	0	1	0	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=			Ajustar sensibilidad		
7	1,5	1	0	1	0	0	0	0			
8	1,5	0	1	0	1	0	0	0			
9	1,5	1	0	0	1	0	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=			Ajustar sensibilidad		
10	2	0	0	0	0	1	0	0			
11	2	0	0	0	0	0	1	0			
12	2	0	0	1	1	0	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=			Ajustar sensibilidad		
13	2,5	1	0	0	0	1	0	0			
14	2,5	0	1	0	0	0	1	0			
15	2,5	0	1	1	1	0	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=			Ajustar sensibilidad		
16	3	0	0	1	0	1	0	0			
17	3	0	0	0	1	0	1	0			
18	3	1	1	0	0	1	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=			Ajustar sensibilidad		
19	3,5	1	0	1	0	1	0	0			
20	3,5	0	1	0	1	0	1	0			
21	3,5	0	1	0	1	1	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=			Ajustar sensibilidad		
22	4	0	0	0	0	1	1	0			
23	4	0	0	1	1	1	0	0			
24	4	1	1	1	0	0	1	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=			Ajustar sensibilidad		
25	4,5	1	0	1	1	0	1	0			
26	4,5	0	1	1	1	0	1	0			
27	4,5	1	0	0	0	1	1	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=			Ajustar sensibilidad		
28	5	0	0	1	0	1	1	0			
29	5	0	0	0	1	1	1	0			
30	5	1	1	0	0	1	1	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=			Ajustar sensibilidad		
31	5	0	0	0	0	0	0	1			

DATA RECORDING FORM
Matrix model for instruments with a maximum capacity of 2 g and 20 g.

		Planilla de toma de datos Max.Cap. 2 g, 20 g									1		
		Pesas									Indicación de la balanza		
carga/g		1	1*	2	2*	5	5*	10	10*	20	Cero	Carga	Cero
		Temp/°C=		h relat./%=		Presión. atm./hPa=					Ajustar sensibilidad		
1	20	0	0	0	0	0	0	0	0	1			
2	1	1	0	0	0	0	0	0	0	0			
3	1	0	1	0	0	0	0	0	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=					Ajustar sensibilidad		
4	2	1	1	0	0	0	0	0	0	0			
5	2	0	0	1	0	0	0	0	0	0			
6	2	0	0	0	1	0	0	0	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=					Ajustar sensibilidad		
7	3	1	0	1	0	0	0	0	0	0			
8	3	0	1	0	1	0	0	0	0	0			
9	3	1	0	0	1	0	0	0	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=					Ajustar sensibilidad		
10	5	1	0	1	1	0	0	0	0	0			
11	5	0	1	1	1	0	0	0	0	0			
12	5	0	0	0	0	1	0	0	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=					Ajustar sensibilidad		
13	7	1	1	0	0	1	0	0	0	0			
14	7	1	1	0	0	0	1	0	0	0			
15	7	0	0	1	0	0	1	0	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=					Ajustar sensibilidad		
16	9	1	1	1	0	1	0	0	0	0			
17	9	0	0	1	1	0	1	0	0	0			
18	9	1	1	1	1	1	0	0	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=					Ajustar sensibilidad		
19	10	0	0	0	0	1	1	0	0	0			
20	10	0	0	0	0	0	0	1	0	0			
21	10	0	0	0	0	0	0	0	1	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=					Ajustar sensibilidad		
22	12	1	1	0	0	0	0	1	0	0			
23	12	0	0	1	0	0	0	0	1	0			
24	12	0	0	0	1	0	0	1	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=					Ajustar sensibilidad		
25	15	0	0	0	0	1	0	1	0	0			
26	15	0	0	0	1	1	0	0	1	0			
27	15	0	0	0	0	0	1	1	1	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=					Ajustar sensibilidad		
28	20	0	0	1	0	0	0	1	1	0			
29	20	0	0	0	0	1	1	0	1	0			
30	20	1	0	1	1	1	0	1	0	0			
		Temp/°C=		h relat./%=		Presión. atm./hPa=					Ajustar sensibilidad		
31	20	0	0	0	0	0	0	0	0	1			

13.2 APPENDIX B: SUGGESTIONS FOR AIR DENSITY ESTIMATION

Note: In Appendix B, symbols are T for temperature in K, and t for temperature in °C

13.2.1 Formulas for air density

The most accurate formula for determining air density in most cases is the one recommended by the CIPM [9]⁴. For the purpose of this Guide, the use of less sophisticated formulas that yield slightly less accurate results is sufficient.

13.2.1.1 Simplified version of the CIPM formula, exponential version

Exponential version taken from [5], section E3:

$$\rho_a = \frac{0.348\ 48\ p - 0.009\ hr \exp(0.061t)}{273.15 + t} \quad (13.2.1.1-1)$$

with

ρ_a air density in kg/m³
 p barometric pressure in hPa
 hr relative air humidity in %.
 t air temperature in °C

The formula gives results with $u_{\text{form}}/\rho_a = 2.4 \times 10^{-4}$ under the following environmental conditions (measurement uncertainties of p , hr , and t not included)

$$\begin{aligned} 600\ \text{hPa} &\leq p \leq 1\ 100\ \text{hPa} \\ 20\ \% &\leq hr \leq 80\ \% \\ 15\ \text{°C} &\leq t \leq 27\ \text{°C} \end{aligned}$$

13.2.1.2 Average air density

When the measurement of temperature and barometric pressure is not possible, the average air density of the site can be calculated from the altitude above sea level, as recommended in [5]:

⁴ The recommended temperature and pressure ranges for the application of the CIPM-2007 formula are: $600\ \text{hPa} \leq p \leq 1\ 100\ \text{hPa}$
 $15\ \text{°C} \leq t \leq 27\ \text{°C}$

$$\rho_a = \rho_0 \exp\left(-\frac{\rho_0}{p_0} g h_{\text{SL}}\right) \quad (13.2.1.2-1)$$

with

$$\begin{aligned} p_0 &= 101\,325 \text{ Pa} \\ \rho_0 &= 1.2 \text{ kg m}^{-3} \\ g &= 9.81 \text{ m s}^{-2} \\ h_{\text{SL}} &= \text{altitude above sea level, in m} \end{aligned}$$

13.2.1.3 Variations of the parameters of the air density components

13.2.1.3.1 Barometric pressure

For any location, the maximum variation is $\Delta p = \pm 40$ hPa around the average⁵. Within these limits, the distribution is not rectangular since extreme values occur only once in several years. Considering the typical atmospheric pressure variation, it is realistic to assume a standard uncertainty

$$u_p = 10 \text{ hPa} \quad (13.2.1.3.1-1)$$

The average barometric pressure $p(h_{\text{SL}})$ can be estimated from the altitude h_{SL} in meters above sea level of the location, using the following relation

$$p(h_{\text{SL}}) = p_0 \exp(-h_{\text{SL}} \times 0.000\,12 \text{ m}^{-1}) \quad (13.2.1.3.1-2)$$

with $p_0 = 1\,013.25$ hPa

13.2.1.3.2 Temperature

The possible variation of the temperature $\Delta t = t_{\text{max}} - t_{\text{min}}$ at the place of use of the instrument can be estimated from information that can be easily obtained:

- of the limits mentioned by the customer's experience;
- of the average of readings of the appropriate records;
- of the control instrument setting, when the place is air-conditioned or temperature controlled;

⁵ Example: in Hannover, Germany, the difference between the highest and lowest barometric pressure observed during 20 years was 77.1 hPa (Information from DWD, German Weather Service)

otherwise some criteria should be applied - e.g.:

$17^{\circ}\text{C} \leq t \leq 27^{\circ}\text{C}$ for a closed office or laboratory with windows,
 $\Delta T \leq 5 \text{ K}$ for windowless locations in the center of a building,
 $-10^{\circ}\text{C} \leq t \leq +30^{\circ}\text{C}$ or $\leq +40^{\circ}\text{C}$ for open workshops, factory halls.

As mentioned for barometric pressure, a rectangular distribution is unlikely to occur for open workshops or factory rooms where atmospheric temperature prevails. However, in order to avoid different assumptions for different room situations, it is recommended to assume a rectangular distribution, as follows,

$$u(t) = \Delta t / \sqrt{12} \quad (13.2.1.3.2-1)$$

13.2.1.3.3 Relative humidity

The possible variation of the relative humidity $\Delta hr = hr_{\max} - hr_{\min}$ at the place of use of the instrument can be estimated from information that can be easily obtained:

of the limits mentioned by the customer's experience;
of the average of appropriate log readings;
of the control instrument setting, if the place is air-conditioned;

failing that, some criterion should be applied, e.g.:

$30 \% \leq hr \leq 80 \%$ for an enclosed office or laboratory with windows,
 $\Delta hr \leq 30 \%$ for windowless locations in the center of a building,
 $20 \% \leq hr \leq 80 \%$ for open workshops, factory halls.

It should be kept in mind that at values of $hr < 40 \%$ could influence the result of heavy electrostatic effects, in high resolution instruments at $hr > 60 \%$ could initiate corrosion.

As mentioned for barometric pressure, a rectangular distribution is not very likely to occur for open workshops or factory halls where atmospheric relative humidity prevails. However, in order to avoid different assumptions for different site situations, it is recommended to assume a rectangular distribution, being

$$u(hr) = \Delta hr / \sqrt{12} \quad (13.2.1.3.3-1)$$

13.2.2 Air density uncertainty

The standard uncertainty of the air density $u(\rho_a)/\rho_a$ is calculated as follows

$$\frac{u(\rho_a)}{\rho_a} = \sqrt{\left(\frac{u_p(\rho_a)}{\rho_a} \cdot u(p)\right)^2 + \left(\frac{u_T(\rho_a)}{\rho_a} \cdot u(T)\right)^2 + \left(\frac{u_{hr}(\rho_a)}{\rho_a} \cdot u(hr)\right)^2 + \left(\frac{u_{\text{form}}(\rho_a)}{\rho_a}\right)^2} \quad (13.2.2-1)$$

with sensitivity coefficients (derived from the CIPM formula [9] for air density)

$$\begin{aligned} u_p(\rho_a)/\rho_a &= 1 \times 10^{-3} \text{ hPa}^{-1} \text{ for barometric pressure} \\ u_T(\rho_a)/\rho_a &= -4 \times 10^{-3} \text{ }^\circ\text{C}^{-1} \text{ for air temperature} \\ u_{hr}(\rho_a)/\rho_a &= -9 \times 10^{-3} \text{ for relative humidity (the unit for } hr \text{ is in this case 1, not \%)} \end{aligned}$$

These sensitivity coefficients can also be used in the equation (13.2.2-1)

Examples of air density standard uncertainty, calculated for different parameters using the simplified formula CIPM-2007, equation (13.2.1.1-1)

$u(p)$ /hPa	Δt /°C	Δh_r	$\frac{u_p(\rho_a)}{\rho_a} u(p)$	$\frac{u_t(\rho_a)}{\rho_a} u(t)$	$\frac{u_{hr}(\rho_a)}{\rho_a} u(hr)$	$\frac{u_{\text{form}}(\rho_a)}{\rho_a}$	$u(\rho_a)/\rho_a$
10	2	0.2	1×10^{-2}	-2.31×10^{-3}	-5.20×10^{-4}	2.40×10^{-4}	1.03×10^{-2}
10	2	1	1×10^{-2}	-2.31×10^{-3}	-2.60×10^{-3}	2.40×10^{-4}	1.06×10^{-2}
10	5	0.2	1×10^{-2}	-5.77×10^{-3}	-5.20×10^{-4}	2.40×10^{-4}	1.16×10^{-2}
10	5	1	1×10^{-2}	-5.77×10^{-3}	-2.60×10^{-3}	2.40×10^{-4}	1.18×10^{-2}
10	10	0.2	1×10^{-2}	-1.15×10^{-2}	-5.20×10^{-4}	2.40×10^{-4}	1.53×10^{-2}
10	10	1	1×10^{-2}	-1.15×10^{-2}	-2.60×10^{-3}	2.40×10^{-4}	1.55×10^{-2}
10	20	0.2	1×10^{-2}	-2.31×10^{-2}	-5.20×10^{-4}	2.40×10^{-4}	2.52×10^{-2}
10	20	1	1×10^{-2}	-2.31×10^{-2}	-2.60×10^{-3}	2.40×10^{-4}	2.53×10^{-2}
10	30	0.2	1×10^{-2}	-3.46×10^{-2}	-5.20×10^{-4}	2.40×10^{-4}	3.61×10^{-2}
10	30	1	1×10^{-2}	-3.46×10^{-2}	-2.60×10^{-3}	2.40×10^{-4}	3.61×10^{-2}
10	40	0.2	1×10^{-2}	-4.62×10^{-2}	-5.20×10^{-4}	2.40×10^{-4}	4.73×10^{-2}
10	40	1	1×10^{-2}	-4.62×10^{-2}	-2.60×10^{-3}	2.40×10^{-4}	4.73×10^{-2}
10	50	0.2	1×10^{-2}	-5.77×10^{-2}	-5.20×10^{-4}	2.40×10^{-4}	5.86×10^{-2}
10	50	1	1×10^{-2}	-5.77×10^{-2}	-2.60×10^{-3}	2.40×10^{-4}	5.87×10^{-2}

13.3 APPENDIX C: COVERAGE FACTOR k FOR EXPANDED MEASUREMENT UNCERTAINTY

Note: in this appendix the general symbol y is used for the result of measurement, not a particular quantity as an indication, an error, a mass of a weighed object, etc.

13.3.1 Objective

The coverage factor k shall in all cases be chosen such that the expanded uncertainty of measurement has a coverage probability of 95.45 %.

13.3.2 Normal distribution and sufficient reliability

The value $k = 2$, corresponds to a 95.45 % probability, applies where

- a) a normal (Gaussian) distribution can be attributed to the error of indication, and
- b) the standard uncertainty $u(E)$ is of sufficient reliability (i.e. it has a sufficient number of degrees of freedom), see JCGM 100 [1].

Normal distribution may be assumed where several (i.e. $N \geq 3$) uncertainty components, each derived from “well-behaved” distributions (normal, rectangular or the like), contribute to $u(E)$ in comparable amounts.

Sufficient reliability is depending on the degrees of freedom. This criterion is met where no Type A contribution to $u(E)$ is based on less than 10 observations. A typical Type A contribution stems from repeatability. Consequently, if during a repeatability test a load is applied not less than 10 times, sufficient reliability can be assumed.

13.3.3 Normal distribution, no sufficient reliability

Where a normal distribution can be attributed to the error of indication, but $u(E)$ is not sufficiently reliable, then the effective degrees of freedom ν_{eff} have to be determined using the Welch-Satterthwaite formula

$$\nu_{\text{eff}} = \frac{u^4(E)}{\sum_{i=1}^N \frac{u_i^4(E)}{\nu_i}} \quad (13.3.3-1)$$

where $u_i(E)$ are the contributions to the standard uncertainty as per (10-2), and v_i is the degrees of freedom of the standard uncertainty contribution $u_i(E)$. Based on v_{eff} the applicable coverage factor k is read from the extended table of [1], Table G.2 or the underlying t -distribution described in [1], Annex C.3.8 may be used to determine the coverage factor k .

13.3.4 Determining k for non-normal distributions considered as normal

In any of the following cases, the expanded uncertainty is $U(y) = ku(y)$.

It may be obvious in a given situation that $u(y)$ contains a Type B uncertainty component of $u_1(y)$ from a contribution whose distribution is non-normal but, e.g., rectangular or triangular, which is significantly greater than all the remaining components. In such a case, $u(y)$ is split up in the (possibly dominant) part u_1 and $u_R = \text{square root of } \sum u_j^2 \text{ with } j \geq 2$, the combined standard uncertainty comprising the remaining contributions, see [1].

If $u_R \leq 0.3 u_1$, then u_1 is considered to be "dominant" and the distribution of y is considered to be essentially identical with that of the dominant contribution.

The coverage factor is chosen according to the shape of the distribution of the dominant component:

for trapezoidal distribution with $\beta < 0.95$.
($\beta = \text{edge parameter, ratio of smaller to larger edge of trapezoid}$)

$$k = \left\{ 1 - \sqrt{[0.05(1 - \beta^2)]} \right\} / \sqrt{[(1 + \beta^2)/6]} \quad (13.3.4-1)$$

for a rectangular distribution ($\beta = 1$): $k = 1.65$,

for a triangular distribution ($\beta = 0$): $k = 1.90$,

for a U-shaped distribution: $k = 1.41$.

The dominant component may itself be composed of 2 dominant components $u_1(y)$, $u_2(y)$, e.g. 2 rectangular making up one trapezoid, in which case u_R will be determined from the remaining u_j with $j \geq 3$.

13.4 APPENDIX D: TERMS

For the purposes of this guide, the terms "internal sensitivity adjustment" and "external sensitivity adjustment" are assumed according to the following definitions:

Internal sensitivity adjustment: It is performed when the weighing instrument has an internal weight with which the sensitivity adjustment is made.

External sensitivity adjustment: This is performed when the balance does not have an internal weight, but the user has an external weight of the required class, with which the adjustment is made. The identification of this weight must be stated on the calibration certificate.

