

General European OMCL Network (GEON)

QUALITY MANAGEMENT DOCUMENT

PA/PH/OMCL (12) 77 R12

QUALIFICATION OF EQUIPMENT

QUALIFICATION OF BALANCES

Full document title and reference	Qualification of Balances - Annex 8 to the OMCL Network Guideline "Qualification of Equipment" <i>PA/PH/OMCL (12) 77 R12</i>
Document type	Guideline
Legislative basis	Council Directive 2001/83/EC and 2001/82/EC, as amended
Date of first adoption	August 2013
Date of original entry into force	December 2013
Date of entry into force of revised document	March 2023
Previous titles/other references / last valid version	<i>PA/PH/OMCL (12) 77 R11</i>
Custodian Organisation	The present document was elaborated by the OMCL Network / EDQM of the Council of Europe
Concerned Network	GEON

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ANNEX 8 OF THE OMCL NETWORK GUIDELINE “QUALIFICATION OF EQUIPMENT”

QUALIFICATION OF BALANCES

Note: Mandatory requirements in this guideline and its annexes are defined using the terms “shall” or “must”. The use of “should” indicates a recommendation. For these parts of the text appropriately justified deviations are acceptable provided they are traceable. The term “can” indicates a possibility or an example with non-binding character.

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INTRODUCTION

This document is the 8th Annex to the core document “Qualification of Equipment”, and it should be used in combination with it when planning, performing and documenting the qualification process of balances.

In addition to this guideline, laboratories shall fulfil the requirements of the Ph. Eur. General Chapter 2.1.7. “Balances for Analytical Purposes”, whenever applicable.

The core document “Qualification of Equipment” contains the general introduction and Levels I and II of qualification, common to all types of instruments. The present Annex 8 contains a general introduction and requirements for balances (electronic - digital) used in OMCLs for physico-chemical and biological tests (Table 1).

Table 1 - Types of balances considered in this guideline:

Ordinary balance name	Resolution
Ultra - Micro Balances	0.1 µg
Micro Balances	1 µg
Semi-micro Balances	0.01 mg

Ordinary balance name	Resolution
Analytical Balances	0.1 mg
Precision Balances	100 - 1 mg
Technical Balances	1 g - 0.1 g

The classification is based on technical data from several manufacturers of balances.

Level III and IV qualifications must be carried out, being an ISO 17025 requirement [1].

Requirements (i.e. parameters to be checked) given in bold should be applied; however, other appropriately justified approaches are acceptable. Level IV exemplary procedures provided in this document have non-binding character. They can be helpful when carrying out the required qualification. Nevertheless, it is left to the professional judgement and background experience of each OMCL to decide on the most relevant procedures to be undertaken in order to give evidence that their balances are working properly and are suitable for their intended use.

If the qualification is performed by the OMCL, the operators shall have the necessary technical expertise to take into account the environmental conditions or any other parameter that may influence the result of the calibration and its associated uncertainty.

GENERAL RECOMMENDATIONS

1) Environmental conditions

The location (room, bench, ...) and the conditions (temperature, relative humidity, barometric pressure, electrostatic and magnetic properties, airflow, vibrations, dust, direct sunlight ...) in which the balance are used should be appropriate for the type of balance, in accordance with the manufacturer's specification, and documented. Level III qualification shall be performed under monitored environmental conditions which represent the operational working conditions (normal conditions of use). Records of monitored environmental conditions that may affect the calibration operation (temperature and optionally relative humidity and barometric pressure) shall be included in the certificate.

If additional devices (e.g. printers, computers) are used for recording indications of the weighing instruments they shall be clearly identified by the user and documented.

2) General set-up - before use/calibration of the balance

- a) Visual inspection of the balance (identification, labelling, contamination, damage, levelling) in order to ensure the condition of the instrument is suitable;
- b) Warming-up the balance for a minimum time as specified by the manufacturer;
- c) Adjustment with external or built-in weights (for instruments intended to be adjusted before use).

3) Selection of weights (test loads) for qualification of balances

The selection of the weights (test loads) should be in accordance with the relevant OIML recommendations [2]. Level III qualification shall be performed using standard weights; however for tests of a comparative nature (e.g. repeatability and eccentricity) and for level IV qualification other test loads may be used.

Standard weights used for calibration of balances shall be calibrated by a provider that is accredited according to ISO 17025. The maintenance of the metrological properties of standard weights shall be ensured by periodical re-calibration and/or verification of documents. The

international recommendation OIML R 111-1 specifies requirements for the standard weights, depending on their mass and accuracy class:

- *metrological requirements* (maximum permissible errors on verification/inspection, expanded uncertainty and conventional mass);
- *technical requirements* (shape, structure, material, magnetic properties, density, surface conditions, adjustment, marking and presentation);
- *requirements for metrological control* (calibration, re-calibration and verification).

Other test loads should be made of a material, shape and structure that allows easy handling, suitable positioning on the balance. Their mass must remain constant over the full period of use for calibration.

Each test load (standard weights and other test loads) shall be:

- uniquely identified;
- clean and, if necessary, wiped according to the manual's instruction or according to OIML R 111-1 recommendations;
- handled with care: forceps/tweezers should be used for smaller weights and cotton gloves for larger weights and they are not to be touched with bare hands.

The class of the weights used in the procedure for calibration of balances shall be chosen depending on the Maximum Permissible Error (MPE) of the balance. MPE of the balance is defined by the laboratory during the initial verification of the balance according to OIML R 76-1 Chapter 3.5 [3], on the basis of the intended use and type of the balance (accuracy class) and standard weights and according to the uncertainty of weighing targeted by the laboratory. The value of the MPE can be changed depending on the uncertainty of measurement estimated during the subsequent calibrations, however it must not exceed two times the value of the MPE established at the initial verification.

According to Euramet, the maximum permissible errors or the uncertainties of calibration of the standard weights shall be compatible with the scale interval d of the instrument and/or the needs of the client regarding uncertainty of calibration of the instrument [4].

If selection of the weights for calibration of the balance is based on nominal value of the mass of the standard weight used for calibration, then the MPE of the weight (Table 2) shall not be greater than the MPE of the balance divided by 3:

$$\text{MPE Weight} \leq \frac{\text{MPE balance}}{3}$$

If selection of weights is made using conventional mass of the standard weight given in the calibration certificate of the standard weight, then the expanded uncertainty of calibration of the weight (U_{CW} , stated in the calibration certificate) shall not be greater than the MPE of the balance divided by 3:

$$U_{CW} \leq \frac{\text{MPE}_{\text{balance}}}{3}$$

Table 2 Maximum permissible errors for weights ($\pm\delta_m$ in mg)

Maximum permissible errors for weights ($\pm \delta m$ in mg)									
Nominal value*	Class E ₁	Class E ₂	Class F ₁	Class F ₂	Class M ₁	Class M ₁₋₂	Class M ₂	Class M ₂₋₃	Class M ₃
5 000 kg			25 000	80 000	250 000	500 000	800 000	1 600 000	2 500 000
2 000 kg			10 000	30 000	100 000	200 000	300 000	600 000	1 000 000
1 000 kg		1 600	5 000	16 000	50 000	100 000	160 000	300 000	500 000
500 kg		800	2 500	8 000	25 000	50 000	80 000	160 000	250 000
200 kg		300	1 000	3 000	10 000	20 000	30 000	60 000	100 000
100 kg		160	500	1 600	5 000	10 000	16 000	30 000	50 000
50 kg	25	80	250	800	2 500	5 000	8 000	16 000	25 000
20 kg	10	30	100	300	1 000		3 000		10 000
10 kg	5.0	16	50	160	500		1 600		5 000
5 kg	2.5	8.0	25	80	250		800		2 500
2 kg	1.0	3.0	10	30	100		300		1 000
1 kg	0.5	1.6	5.0	16	50		160		500
500 g	0.25	0.8	2.5	8.0	25		80		250
200 g	0.10	0.3	1.0	3.0	10		30		100
100 g	0.05	0.16	0.5	1.6	5.0		16		50
50 g	0.03	0.10	0.3	1.0	3.0		10		30
20 g	0.025	0.08	0.25	0.8	2.5		8.0		25
10 g	0.020	0.06	0.20	0.6	2.0		6.0		20
5 g	0.016	0.05	0.16	0.5	1.6		5.0		16
2 g	0.012	0.04	0.12	0.4	1.2		4.0		12
1 g	0.010	0.03	0.10	0.3	1.0		3.0		10
500 mg	0.008	0.025	0.08	0.25	0.8		2.5		
200 mg	0.006	0.020	0.06	0.20	0.6		2.0		
100 mg	0.005	0.016	0.05	0.16	0.5		1.6		
50 mg	0.004	0.012	0.04	0.12	0.4				
20 mg	0.003	0.010	0.03	0.10	0.3				
10 mg	0.003	0.008	0.025	0.08	0.25				
5 mg	0.003	0.006	0.020	0.06	0.20				
2 mg	0.003	0.006	0.020	0.06	0.20				
1 mg	0.003	0.006	0.020	0.06	0.20				

Refer to OIML R 111-1 for further details related to the classes [2].

Level III. Periodic and motivated instrument checks
Typical requirements for qualification of balances (Calibration and Verification)

QUALIFICATION

Qualification of balances encompasses calibration and verification (or declaration of conformity).

Level III qualification shall be performed using certified calibrated weights; however for tests of a comparative nature (e.g. repeatability and eccentricity) other test loads may be used.

Balances shall be qualified at the place of use. When balances are installed or moved into a new environment, or after significant repair or maintenance operations, calibration and verification shall be carried out to maintain confidence in the performance of the equipment. The significance of changes (e.g. repairing a balance or transfer to another laboratory) should be evaluated to take the appropriate actions (e.g. calibration only, full re-qualification, no actions) in relation to the impact and risks of these changes. Qualification of balances shall be conducted on a regular basis. The frequency of periodic calibration can be defined on the basis of a risk-based evaluation; an example of the relevant factors affecting calibration frequency is given in Appendix II. However, calibration frequency shall be set according to pre-defined and documented criteria based on sound scientific justifications [5].

Requirements and (if applicable) corresponding typical acceptance criteria are given in Table 3; however, other appropriately justified approaches are acceptable.

Table 3 Requirements and corresponding typical acceptance criteria for Level III qualification of balances

Parameter to be checked	Acceptance criteria
Error of indication	$ E_i \leq \text{MPE balance}$
Repeatability	$E_r \leq \text{MPE balance}$
Eccentricity	$ E_e \leq \text{MPE balance}$

CALIBRATION

The calibration shall comprise testing of at least the following parameters: **error of indication, repeatability and eccentricity.**

1) Error of indication

The test is performed by reading the values of weighing of minimum 5 weights within the operational range or minimum and maximum capacity of the balance or the maximum operational range defined by the laboratory, with one replicate per weight, in increasing and/or decreasing loading mode (if the balance can be used in decreasing mode).

The error of indication is the difference between the nominal or the conventional value (depending on the choice of the laboratory) of the weight and the reading values:

$$E_i = I - m_{ref}$$

where,

I is measured value (Indication)

m_{ref} is the reference value of the mass of the weight used (nominal value or conventional value).

The nominal value of the mass of the weight can be used as reference value for calculation of error of indication if MPE of the weight (Table 2) is not greater than the MPE of the balance divided by 3. If this condition cannot be fulfilled, the conventional value of the mass of the weight (stated in the calibration certificate) shall be used for calculation of error of indication, provided that the expanded uncertainty of calibration of the weight (U_{CW} , stated in the calibration certificate) is not greater than the MPE of the balance divided by 3.

IMPORTANT: in cases where a balance is used for differential weighing (i.e. a standard and a test mass are weighed consecutively on the same weighing pan and the difference in their indicated value forms the basis for the calibration), the error of indication should be determined using the tare.

2) Repeatability

The repeatability of the balance can be assessed using two approaches: a) determination of repeatability error or b) standard deviation.

Approach 1 (Repeatability error):

The test is performed by reading the values of repeated weighing of at least one weight in a range between 50% of maximum capacity or the maximum operational range defined by the laboratory and the maximum capacity of the balance or the maximum operational range defined by the laboratory (80% of maximum capacity is sufficient). Minimum 6 replicates are required, except for technical balances where minimum of 3 replicates are required.

The repeatability error (E_r) is calculated as the difference between the minimum and maximum values obtained.

Approach 2 (Standard deviation)

The test is performed by reading the values of repeated weighing of at least one test load in a range between 50% of maximum capacity and the maximum capacity of the balance or the maximum operational range defined by the laboratory. A minimum of 5 replicates should be carried out for each weight.

Standard deviation is calculated from the repeated measurements:

$$S = \sqrt{\frac{1}{n-1} \sum_{i=1}^n (I_i - \bar{I})^2}$$

n is number of readings

I_i is measured value (Indication)

\bar{I} is average value

$$\bar{I} = \frac{1}{n} \sum_{i=1}^n I_i$$

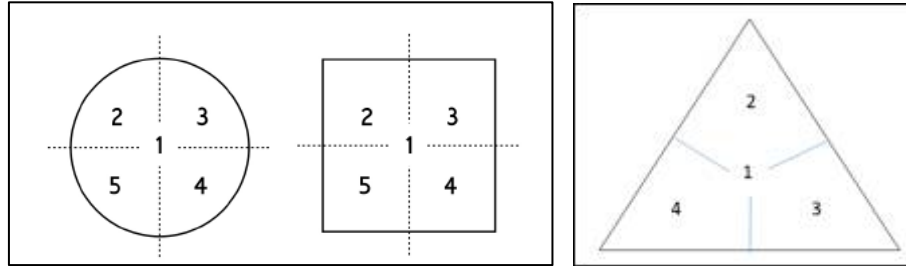
The laboratory can decide to follow a combined procedure to perform error of indication and repeatability tests simultaneously.

3) Eccentricity

The test is performed by reading the values of weighing of the weight at approximately 1/3 of the maximum capacity of the balance or the maximum operational range defined by the laboratory.

The test is performed by placing the weight on the centre of the pan and on the centre of four quadrants, as depicted in figures below. The values of the weight located in the centre and off-centre positions are recorded.

A triangular pane should be divided in three, and the weight placed in the centre and in each corner as indicated in the figure below. If the example spreadsheet is used for calculations, no value is added for position 5.



The eccentricity error (E_e) is calculated as:

- the largest deviation between an off-centre and the centre readings ($|\Delta I_{ecc}|_{max}$) or
- the largest deviation between the nominal value of the weight and the reading values at different positions

MEASUREMENT UNCERTAINTY OF CALIBRATION OF THE BALANCE

Evaluation of the measurement uncertainty of calibration of the balance should take into consideration all the relevant contributors.

Estimation of uncertainty in calibration of the balance encompasses the following steps:

- 1) Specification of the measurand
- 2) Identification of uncertainty sources
- 3) Quantification of uncertainty components for each contributor
- 4) Calculation of combined standard uncertainty and expanded uncertainty

The sources of uncertainty of calibration of the balance (error of indication) may include the uncertainty due to repeatability of the instrument, $u_{(rep)}$, uncertainty of resolution of the balance without load $u_{(d0)}$ and with load $u_{(dt)}$, uncertainty of eccentricity of load $u^2_{(ecc)}$, uncertainty of reference weight $u(m_{ref})$, etc. The combined standard uncertainty of calibration of the balance (u_c) may be calculated from the standard uncertainties of these contributors:

$$u_c^2 = u^2_{(rep)} + u^2_{(d0)} + u^2_{(dt)} + u^2_{(ecc)} + u^2_{(m_{ref})}$$

Expanded uncertainty (U), for $k = 2$, at approximately 95% level of confidence is calculated as:

$$U = 2 \cdot u_c$$

Detailed descriptions of calculation formulas are given in the example in Appendix III.

VERIFICATION

The verification of the balance includes declaration of conformity of results from calibration of the balance to a given specification. The verification results shall be documented according to the OMCL standard operating procedure.

Approach 1. Conformity may be stated if results from each individual test conform to the specified limits:

1. Error on indication

The difference between the results of several weighings of the same load shall not be greater than the absolute value of the maximum permissible error (MPE) of the instrument for that load, i.e. the absolute value of error (E_i) shall not exceed the MPE established for the balance.

$$|E_i| \leq \text{MPE balance}$$

2. Repeatability

The repeatability error shall not exceed the MPE of the balance (this is applicable if approach 1 is used for assessing repeatability of the method):

$$E_r \leq \text{MPE balance}$$

3. Eccentricity

The eccentricity error shall not exceed the MPE of the balance:

$$|E_e| \leq \text{MPE balance}$$

Approach 2. Alternatively, the conformity of the balance may be declared if the following conditions are fulfilled (this is applicable if approach 2 is used for assessing repeatability of the method):

$$|E_i| + U \leq \text{MPE balance}$$

Where the calibration is performed by external providers, the declaration of conformity should be provided upon request or, alternatively, the verification shall be carried out by the OMCL. An example is provided in Appendix I.

REPORTING RESULTS OF QUALIFICATION

The results of the qualification shall be reported according to the requirements given in ISO 17025:2017 [1].

Level IV. In-use instrument checks

Level IV qualification of balances encompasses in-between calibration controls. Typical requirements, acceptance limits and frequencies are given in Table 4.

Table 4 Typical requirements, acceptance limits and frequencies for Level IV qualification of the balances

Parameter to be checked	Acceptance criteria	Frequency
Levelling	The spirit level bubble must be within the target. Electronic balance: confirmation using the levelling indicator.	Every day of use
Automated internal adjustment (self-testing)/external adjustment	N/A	Every day of use
Control with external weights	OMCLs shall define their own acceptance criteria	At least once a week

Additional parameters can be checked during Level IV qualification based on the professional judgement and background experience of each OMCL. In addition, performance checks (Repeatability and Sensitivity) described in Ph. Eur. General Chapter 2.1.7. “Balances for Analytical Purposes” shall be performed, whenever applicable.

1) Levelling

The levelling procedure depends on the type of balance and can be carried out manually or in automatic mode. For electronic balances, use an electronic level indicator.

For example, for the manual check, the spirit level position is checked visually. Appropriate adjustments of the balance should be made if the bubble of the spirit level is not in the centre of the target.

2) Adjustment: automated internal adjustment (self-testing) or by external standard weight

Manufacturer’s instructions should be followed.

3) Control with external weights

Several procedures are possible, for example, periodic checks of the repeatability of weighing results or calculation of the error of indication of the instrument or balance drift evaluation.

For example:

Error on indication: Test is performed by reading of the weight of at least 1 test load (standard weight) within the operational range, with one replicate and comparing to the nominal value of the test load (weight) or with the conventional mass as reference values. Error of indication is calculated using the same formula described under error of indication for calibration and compared to the MPE of the balance (as described in verification).

Repeatability: Test is performed using at least 1 test load (weight) within the operational range, with a minimum of 5 replicates. Calculate standard deviation and compare it to the pre-defined limits based on the standard deviation obtained from the repeatability test performed during

calibration of the balance or from previous repeatability data of the measurements in the laboratory.

The data obtained within a period of time can be visualised in several ways, for example using control charts.

They are two types of control charts:

- 1) statistical control chart, based on standard deviation and the assumption of a normal distribution of the weighing of the control mass on the balance; there is no need to use a calibrated weight;
- 2) metrological control chart, based on the MPE of the balance; in this case a calibrated weight adapted to the MPE of the balance should be used.

The minimum sample weight is provided by the manufacturer; however it should be periodically verified taking into account the standard deviation value obtained in the repeatability test, the uncertainty of measurement and the intended use of the equipment, following the manufacturer's instructions or the EURAMET Calibration Guide 18 [4].

GLOSSARY - TERMS AND DEFINITIONS

Note: synonyms are reported in brackets.

Adjustment: the set of operations carried out on a measuring system so that it provides prescribed indications corresponding to given values of a quantity to be measured [4].

Calibration: an operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication [6].

Conventional mass (conventional value of mass): the conventional value of the result of weighing in air, in accordance with OIML D 28. For a weight taken at a reference temperature (t_{ref}) of 20°C, the conventional mass is the mass of a reference weight of a density (r_{ref}) of 8000 kg m⁻³ which it balances in air of a reference density (r_0) of 1.2 kg m⁻³ [2].

Error of indication (trueness, accuracy): closeness of agreement between the average of an infinite number of replicate measured quantity values and a reference quantity value [6]. When the result is expressed as % of the conventional/nominal mass of the used weight, this parameter is called as “sensitivity” [9].

Maximum Permissible Error: the maximum difference, positive or negative, allowed by regulation between the indication of an instrument and the corresponding true value, as determined by reference standard masses or standard weights, with the instrument being at zero at no-load, in the reference position [3].

Measurement uncertainty: a parameter associated with the result of a measurement that characterises the dispersion of the values that could be reasonably attributed to the measurand [7].

Minimum sample weight: the smallest sample quantity weighed on a balance that still satisfies a pre-defined weighing accuracy requirement [4].

Nominal value of the weight (**Nominal quantity value**): rounded or approximate value of a characterising quantity of a measuring instrument or measuring system that provides guidance for its appropriate use [6].

Readability or scale interval (d): Value expressed in units of mass of: the difference between the values corresponding to two consecutive scale marks, for analogue indication, or the difference between two consecutive indicated values, for digital indication [3].

Repeatability: measurement precision under a set of repeatability conditions of measurement [6].

Repeatability condition of measurement (repeatability condition): is a condition of measurement, out of a set of conditions that includes the same measurement procedure, same operators, same measuring system, same operating conditions and same location, and replicate measurements on the same or similar objects over a short period of time [6].

Resolution: smallest change in a quantity being measured that causes a perceptible change in the corresponding indication [6].

Verification: provision of objective evidence that a given item fulfils specified requirements. [6].

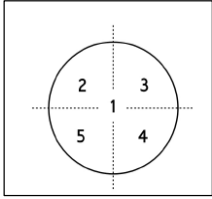
Weight: the material measure of mass, regulated in regard to its physical and metrological characteristics: shape, dimensions, material, surface quality, nominal value, density, magnetic properties and maximum permissible error [2].

REFERENCES

(For all references, the latest version applies)

1. ISO/IEC 17025 “General requirements for the competence of testing and calibration laboratories”
2. OIML R 111-1: 2004 (E) Weights of classes E1, E2, F1, F2, M1, M1–2, M2, M2–3 and M3 Part 1: Metrological and technical requirements
3. OIML R 76-1 Nonautomatic weighing instruments Part 1: Metrological and technical requirements - Tests
4. ILAC G24 2007 (OIML D10 2007) Guidelines for the determination of calibration intervals of measuring instruments
5. JCGM 200:2012 International vocabulary of metrology – Basic and general concepts and associated terms (VIM)
6. Evaluation of measurement data – Guide to the expression of uncertainty in measurement (GUM), JCGM 100: 2008
7. EURAMET Calibration Guide No. 18 Version 4.0 (11/2015), Guidelines on the Calibration of Non-Automatic Weighing Instruments
8. OMCL Guideline Measurement Uncertainty - Annex 1.1. Estimation of the measurement uncertainty of concentration of solutions prepared in laboratory PA/PH/OMCL (18) 146 R1 CORR
9. Ph. Eur. 2.1.7 “Balances for Analytical Purposes”.

Appendix I - Example of verification report

Level III Periodic and motivated instrument check					
BALANCE					
Brand: Sartorius			Model: BP221S		
Batch number: 81203905			Identification number		
Maximum capacity:	220g	Resolution		0.1mg	
		Readability/scale interval		0.0001g	
USED WEIGHTS					
Standard weights class E2 (certificate number: Z16 06033/06035; date of calibration: 12.05.2019)					
ENVIRONMENTAL CONDITIONS					
Temperature: 21.0 °C at the beginning of calibration ($\Delta T=0.4^{\circ}\text{C}$)			Vibration: no		
Humidity: 40 % RH			Dust: no		
Barometric pressure: 990 hPa			Air currents: no		
ERROR OF INDICATION (E_i)					
Used weight nominal value (g)	Read value in increasing mode (g)	E_i (mg)	Read value in decreasing mode (g)	E_i (mg)	MPE for the balance (mg)
0.001	0.0010	0.0	0.0010	0.0	0.90
5	5.0000	0.0	5.0000	0.0	0.90
50	50.0000	0.0	50.0000	0.0	0.90
100	100.0001	0.1	100.0001	0.1	0.90
200	200.0003	0.3	200.0003	0.3	0.90
REPEATABILITY ERROR (E_r)					
Used weight nominal value (g)		100g ($\approx 1/2$ maximum capacity)			
Measurement	Read value (g)	Measurement	Read value (g)	E_r (mg)	MPE for the balance (mg)
F1	100.0000	F6	100.0001	0.2	0.90
F2	100.0001	F7	100.0001		
F3	100.0000	F8	100.0001		
F4	100.0000	F9	100.0001		
F5	99.9999	F10	99.9999		
ECCENTRICITY ERROR (E_e)					
Position	Used weight nominal value (g)	Read value (g)	E_e (mg)	MPE for the balance (mg)	Positions 
1	70 ($\approx 1/3$ maximum capacity)	70.0004	0.4	0.90	
2		70.0003	0.3		
3		70.0002	0.2		
4		70.0003	0.3		
5		70.0003	0.3		
Verification result		Date		Signature	
Complies		15.06.2019		Analyst 1	

E_i (error of indication) is conventionally displayed as absolute error

Appendix II - Factors affecting calibration frequency

Examples of factors which may affect the intervals of qualification or in-between qualification are listed below:

- uncertainty of measurement required or declared by the laboratory;
- risk of a measuring instrument exceeding the limits of the maximum permissible error when in use;
- cost of necessary correction measures when it is found that the instrument was not appropriate over a long period of time;
- type and frequency of usage of instrument;
- tendency to wear and drift;
- manufacturer's recommendation;
- extent and severity of use;
- environmental conditions (climatic conditions, vibration, ionising radiation, etc.);
- trend data obtained from previous calibration records;
- recorded history of maintenance and servicing;
- frequency of cross-checking against other reference standards or measuring devices;
- frequency and quality of intermediate checks in the meantime;
- transportation arrangements and risk; and
- degree to which the serving personnel are trained.

Each time a balance is calibrated on a routine basis, the subsequent interval may be extended if it is consistently found to be within the requirements e.g. 80% of the maximum permissible error that is required for measurement. The subsequent interval is reduced if it is found to be outside this maximum permissible error. This "outside" response should produce a rapid adjustment of intervals and is easily carried out without clerical effort.

It would be inappropriate to take excessively extend the interval. The risk associated with withdrawing large numbers of certificates issued may ultimately be unacceptable.

This text is extracted from the ILAC G24 2007 (E), OIML D10 2007(E) Guidelines for the determination of calibration intervals of measuring instruments [4].

Appendix III - Evaluation of uncertainty of calibration of a balance

This appendix gives an example to demonstrate the procedure for evaluating uncertainty of measurement in calibration of the balance.

When using the balance for normal usage, the conditions (i.e. weighing process, environment, repeatability, loads, etc.) are different than during calibration. Therefore, the uncertainty of the daily usage of the balance (uncertainty of the weighing result) is different than the uncertainty of calibration of the balance and it is estimated using the uncertainty of calibration of the balance and other relevant contributors: uncertainty of environmental conditions and uncertainty from operation of the instrument (weight to weight variability, taken from repeated weighing - repeatability). An example of calculation of uncertainty of the weighing result is described in Annex 1 of the guideline Uncertainty of measurement [8].

Example:

Calibration of electronic balance (maximum weighing capacity 220 g/scale interval, $d = 0.0001$ g)

Conditions specific for calibration:

Temperature: at the beginning of calibration: 20°C ($\Delta T = 0.4^{\circ}\text{C}$)

Relative humidity: 42.7 ± 1.4 % RH

Barometric pressure: 992 ± 6 hPa

Standard weight (E2): Nominal value of the weight: 100 g, Conventional mass: 100.000066 g, $U_{m_c} = 0.000050$ g ($k = 2$, for approximately 95% level of confidence).

The obtained calibration results are given in Table 1 (Appendix III). Please note that the value of the average below reported is rounded according to the standard deviation obtained.

1. Repeatability		
	<i>Result (indication) (g)</i>	
<i>Test load 100 g</i>		
1	100.0001	
2	100.0001	
3	100.0002	
4	100.0001	
5	100.0001	
6	100.0001	
7	100.0001	
8	100.0001	
9	99.9999	
10	100.0001	
Average	100.00009	
Standard deviation $S(I_j)$	7.37865E-05	
2. Eccentricity		
<i>Position</i>	<i>Indication (g)</i>	<i>Error ($\Delta lecc$)(g)</i>
1. centre	100.0000	0.0000
2. left forward	100.0000	0.0000
3. left back	100.0003	0.0003
4. right back	100.0002	0.0002
5. right forward	100.0003	0.0003
$\Delta lecc$ max	0.0003	

1. Step 1. Specification of a measurand

The measurand is error of indication (E_i):

$$E_i = I - m_{ref}$$

where,

I is the measured value (Indication)

m_{ref} is the reference value of the mass of the weight used (nominal value or conventional value).

2. Step 2. Identification of uncertainty sources

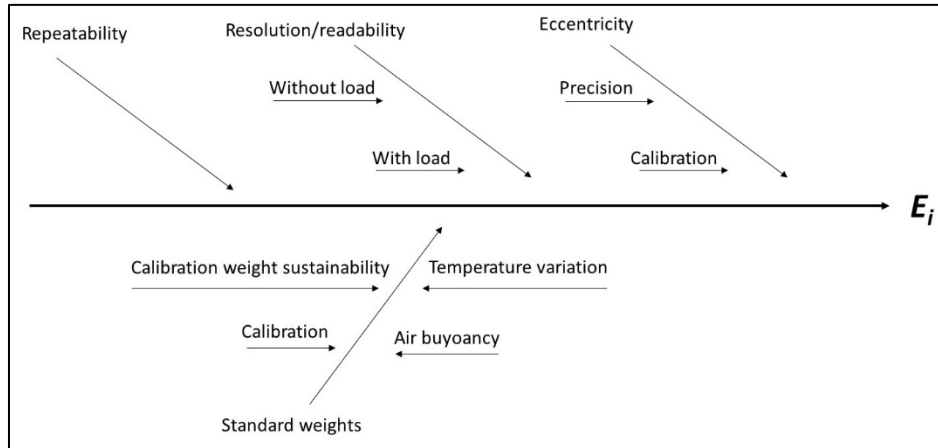


Fig. 1 Cause and effect diagram

3. Step 3. Quantification of uncertainty components

3.1 Component 1. Uncertainty of Repeatability of the instrument, $u_{(rep)}$

The repeatability test can be performed by reading/recording of results (indications, I_j) of repeated depositions of a single test load or using different test loads. The result (indication, I_j) may be recorded (one value) for each individual deposition of the load obtained during the repeatability test or the mean value (repeated measurements) for each individual deposition of the load obtained during the error of indication test.

Where only one repeatability test is performed, using a single test load, the uncertainty of repeatability can be considered as representative for the whole range of the instrument (normal distribution is assumed):

$$u_{(rep)} = S(I_j)$$

Where, $S(I_j)$ = Standard deviation calculated from repeatability test of the balance

Therefore:

$$u_{(rep)} = 0.0000737865 \text{ g}$$

If the result (indication) is the mean of repeated measurements (n) for each individual deposition of the same test load, during the error of indication test, the corresponding standard uncertainty is:

$$u_{(rep)} = \frac{S(I_j)}{\sqrt{n}}$$

If different test loads are used, several values for $S(I_j)$ are obtained. In this case, the greater value of $S(I_j)$ should be used as the uncertainty of repeatability of instrument.

3.2 Component 2. Uncertainty related to resolution of the balance

The uncertainty related to resolution of the balance is linked to rounding of a zero indication after a zero-setting or tare balancing, and rounding of an on-load indication after deposition of the load.

Uncertainty related to resolution (d_0) of the balance without load (u_{d_0}) is calculated as (rectangular or uniform distribution is assumed):

$$u_{(d_0)} = \frac{d_0}{2\sqrt{3}}$$

$$u_{(d_0)} = \frac{0.0001g}{2\sqrt{3}} = 0.000029g$$

Uncertainty related to resolution (d_t) of the balance with load (u_{d_t}) is calculated as (rectangular or uniform distribution is assumed):

$$u_{(d_t)} = \frac{d_t}{2\sqrt{3}}$$

$$u_{(d_t)} = \frac{0.0001g}{2\sqrt{3}} = 0.000029g$$

3.3 Component 3. Uncertainty due to eccentricity of load, $u_{(ecc)}$

This uncertainty accounts for the error due to the position of the test load around the centre of gravity of the balance or due to the use of more than one weight as a test load. This effect is usually neglected, but in some cases it should be taken into account. Considering that maximum eccentricity is the largest difference, the uncertainty may be calculated as (assuming rectangular distribution):

$$u_{(ecc)} = \frac{I|\Delta I_{ecc}|_{max}}{2 \cdot L_{ecc}\sqrt{3}}$$

where:

I is indication

ΔI_{ecc} is maximum eccentricity

L_{ecc} is the test load

Using the results from the obtained calibration (Table 1, appendix III) the calculated value for uncertainty due to eccentricity of load, $u_{rel(ecc)}$ is:

$$u_{(ecc)} = \frac{100.0003 \cdot 0.0003}{2 \cdot 100\sqrt{3}} = 0.0000866 \text{ g}$$

The Standard uncertainty of indication $u(I)$ may be calculated as:

$$u^2(I) = u^2_{(rep)} + u^2_{(d_0)} + u^2_{(d_t)} + u^2_{(ecc)}$$

$$u^2(I) = 0.0000737865^2 + 0.000029^2 + 0.000029^2 + 0.0000866^2$$

$$u(I) = \sqrt{0.0000737865^2 + 0.000029^2 + 0.000029^2 + 0.0000866^2} = 0.000121g$$

3.4 Component 4. Uncertainty of the reference weight ($u(m_{ref})$)

3.4.1 Standard uncertainty of conversion of nominal value of the weight to conventional value of mass (m_c), $u(m_c)$

The conventional value of mass, expanded uncertainty U_{m_c} and the coverage factor k are given in the calibration certificate of the reference weights used for calibration of the balance.

$$u_{m_c} = \frac{U_{m_c}}{2}$$

where:

U_{m_c} is the expanded uncertainty of calibration (for $k = 2$ at approximately 95% level of confidence) given in the certificate of calibration of the weight used

Therefore:

$$u_{m_c} = \frac{0.00005g}{2} = 0.000025g$$

When the standard weight is used with its nominal value of the weight (rectangular distribution is assumed):

$$u_{m_c} = \frac{MPE}{\sqrt{3}}$$

MPE is maximum permissible error given in OIML R111.

MPE for Class E2 weights of 100 g is 0.00016 g (OIML R111). Therefore:

$$u_{m_c} = \frac{0.00016}{\sqrt{3}} = 0.000092376 \text{ g}$$

3.4.2. Uncertainty due to the possible drift of the m_c since last calibration (Calibration weight sustainability) $u(m_D)$ -

If data trend shows significant drift of the weight over time (greater than the uncertainty of calibration of the weight):

$$u(m_D) = \frac{D_{max}}{\sqrt{3}}$$

The value of D_{max} is assumed based on the difference in m_c from consecutive calibration certificates of the standard weights. In the absence of information on drift, the value of D_{max} is the MPE according to OIML R111.

For standard weights of 100 g (class E2), $D_{max} = 0.00016$ g. Therefore:

$$u(m_D) = \frac{0.00016}{\sqrt{3}} = 0.000092376 \text{ g}$$

If there are only one or two calibrations of the weights, D_{max} is not known. In this case, it is recommended to take: $u(m_D) = u(m_c)$

3.4.3. Uncertainty due to temperature difference $u(m_{temp})$

Temperature variations can affect the response of the balance. The effect depends on the mass of the weight (m), variations of temperature during the calibration (ΔT) and on the sensitivity of the

balance to temperature changes (C , provided by the manufacturer). The uncertainty can be calculated as follows:

$$u(m_{temp}) = \frac{C \times \Delta T \times m}{\sqrt{3}}$$

Or alternatively:

$$u(m_{temp}) = \frac{\Delta m}{\sqrt{3}}$$

Δm -depends on the known temperature difference ΔT and mass of the standard weight (m), and is given in Appendix F, EURAMET calibration guide, 18, version 4.0.

This effect is relevant for weights of classes F1 and higher. Usually the weights are acclimatised according to Appendix F, EURAMET calibration guide, 18, version 4.0, so the effect of convection can be neglected in the estimation of combined uncertainty of calibration.

Standard uncertainty of reference weight ($u^2(m_{ref})$)

$$u^2(m_{ref}) = u^2(m_c) + u^2(m_D) + u^2(m_{temp})$$

If $u^2(m_{temp})$ is neglected, then:

$$u^2(m_{ref}) = \sqrt{u^2(m_c) + u^2(m_D)}$$

$$u(m_{ref}) = \sqrt{0.000025^2 + 0.000092376^2} = \sqrt{0.0000000092} \text{ g}$$

$$u(m_{ref}) = 0.0000959 \text{ g}$$

4. Step 4. Calculation of Combined standard uncertainty and expanded uncertainty of the calibration of balance

The combined standard uncertainty of calibration of the balance u_c is:

$$u_c^2 = u^2(I) + u^2(m_{ref})$$

$u^2(I)$ - standard uncertainty of indication

$u^2(m_{ref})$ - standard uncertainty of reference weight

Therefore:

$$u_c = \sqrt{u^2(I) + u^2(m_{ref})} = \sqrt{0.000121^2 + 0.0000959^2} = 0.000154 \text{ g}$$

Expanded uncertainty (U), for $k = 2$, at approximately 95% level of confidence is:

$$U = 2 \cdot u_c = 0.00031 \text{ g}$$

5. Step 5. Reporting of results

The calibration report/certificate shall state the value of expanded uncertainty, together with the coverage factor (k) and statement of coverage probability:

$$U = 0.00031 \text{ g} \text{ (} k = 2, \text{ for approximately 95\% level of confidence)}$$