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Guidelines on the Calibration of Automatic Gravimetric Filling Instruments
DRAFT CALIBRATION METHOD

CONTENTS

1 INTRODUCTION

Automatic gravimetric filling instruments [1] are widely used to fill containers with predetermined and virtually constant mass of product from bulk by automatic weighing. While for some applications specified by law, automatic gravimetric filling instruments may be subject to legal metrological control, there is a growing need for confirmation of their metrological characteristics by calibration, eg. where required by EN ISO 9001 or EN ISO/IEC 17025 standards.

2 SCOPE

This document contains guidance for the calibration of automatic gravimetric filling instruments (hereafter called "instrument"), in particular for

1. measurements to be performed,
2. determination of the conventional mass of the test fills [1] and container(s),
3. calculation of the measuring results,
4. evaluation of the uncertainty of measurement,
5. contents of the calibration certificates.

The guide refers to automatic operation of the instrument.

The calibration of the instruments in the static mode (non-automatic operation) in accordance with EURAMET Calibration Guide No. 18 [2] is not representative one for the actual operation of the instrument.

The object of the calibration is the preset value [1], i.e. value, expressed in units of mass, preset by the operator, in order to define the nominal value of the fills. The actual value of the fill will be affected by dynamic effects, the load type and properties, the container properties, local gravity, the load temperature and density, and the temperature and density of the surrounding air.

The uncertainty of measurement depends significantly on properties of the calibrated instrument itself, the control instrument, the characteristics of the test loads, the container, not only on the equipment of the calibrating laboratory; it can to some extent be reduced by increasing the number of measurements performed for a calibration. This guideline does not specify lower or upper boundaries for the uncertainty of measurement.

It is up to the calibrating laboratory and the client to agree on the anticipated value of the uncertainty of measurement, which is appropriate in view of the use of the instrument and in view of the cost of the calibration.

While it is not intended to present one or few uniform procedures the use of which would be obligatory, this document gives general guidance for establishing of calibration procedures the results of which may be considered as equivalent within the EURAMET Member Organisations.

Any such procedure must include, for a limited number of the preset values, the determination of the preset value error and the uncertainty of measurement assigned to these errors. The test procedure should as closely as possible resemble the weighing operations that are routinely being performed by the user – e.g. production of fills with nominal values and material, which are actualty used by client.

The information presented in this guideline is intended to serve, and should be observed by

1. bodies accrediting laboratories for the calibration of automatic gravimetric filling instruments,
2. laboratories accredited for the calibration of automatic gravimetric filling instruments,
3. manufacturers and other users using calibrated automatic gravimetric filling instruments for measurements relevant for the quality of production subject to QM requirements (e.g. EN ISO 9000 series, EN ISO 10012, EN ISO/IEC 17025).

3 TERMINOLOGY AND SYMBOLS

The terminology used in this document is mainly based on existing documents

- OIML R61-1 [1] for terms related to the operation, construction and metrological characterization of automatic catchweighing instruments
- EURAMET Calibration Guide No. 18 [2] for terms related with the static weighing and calibration of control instrument,

Such terms are not explained in this document, but where they first appear, references will be indicated.

Symbols whose meanings are not self-evident, will be explained where they are first used. Those that are used in more than one section are collected in Activity 1.3.4.

4 GENERAL ASPECTS OF THE CALIBRATION

4.1 Elements of the calibration

Calibration consists of

1. determining the conventional mass of the containers,
2. applying material tests to the instrument under specified conditions,
3. determining the preset value error at dynamic weighing,
4. estimating the uncertainty of measurement to be attributed to the results.

4.1.1 Range of calibration

Unless requested otherwise by the client, the calibration extends over the range limited by the minimum nominal value of fills produced on-site Min' and the largest nominal value of fills produced on-site Max'.

The calibration is performed at the rate operation requested and specified in advance by the client. Normally these conditions are the same as conditions during the actual weighing process.

The calibration it is only valid for these conditions.

4.1.2 Place of calibration

Calibration is performed in the location where the instrument is being installed. The calibration is valid for the place of installation.

4.1.3 Preconditions, preparations

The calibration should be done under normal conditions of use (air currents, vibrations, stability of the weighing site etc.) of the instrument. The calibration should not be performed unless

1. the instrument can be readily identified,
2. all functions of the instrument are free from effects of contamination or damage, and functions essential for the calibration operate as intended,
3. presentation of preset values is unambiguous and indications, where given, are easily readable,
4. the instrument is levelled, if applicable,
5. the instrument shall be operated and fills produces for a time period under normal operating conditions to enable stability.

If agreed with the client, the instrument could be statically adjusted before the calibration. Adjustment should be performed with the means that are normally applied by the client, and following the manufacturer's instructions where available.

Instruments fitted with an automatic zero-setting device or a zero-tracking device [1] should be calibrated with the device operative or not, as set by the client.

The user of the instrument should be asked to ensure that the normal conditions of use prevail during the calibration. In this way disturbing effects such as air currents, vibrations, or inclination of the measuring platform will, so far as is possible, be inherent in the measured values and will therefore be included in the determined uncertainty of measurement.

4.2 Control instrument

A control instrument [1] is used to determine the conventional mass of test fills in containers and the conventional mass of empty containers.

The control instrument may be either separate (a weighing instrument other than the instrument being calibrated) or integral (when a static weighing mode is provided by the instrument being calibrated).

The control instrument, whether separate or integral, should ensure the determination of the conventional mass of each test fill to an accuracy, which is equal or better than the expected uncertainty of calibration of the calibrated instrument.

Generally, the control instrument should have a resolution better than the calibrated instrument and, if applicable, ensure the determination of the conventional mass of each test load to an accuracy of at least one-third of appropriate tolerances for the calibrated instrument defined by client.

In some cases, it may be necessary to use one control instrument for determination of the conventional mass of test fills in containers and another for determination of the conventional mass of empty containers.

Details on determination of the conventional mass of the containers and test fills and its uncertainty on the control instrument are given in the section 4.4 and 7.1.

4.3 Containers

The same kind of containers need to be used during the calibration as they are used during the production process.

4.4 Test fills

The test fills should be made of the type of product, which is normally weighed on the calibrated instrument. For the purpose of calibration, their traceability to the SI unit of mass shall be demonstrated.

The mass of test fills must remain constant throughout the period in which they are used for calibration.

The conventional mass of the test fills is normally determined at the time and place of calibration of the instrument.

The necessary measurements to determine the conventional mass of the test fills are performed on the control instrument. The procedure requires measurements of empty containers (i.e. a tare value) and measurements of filled containers (i.e. a gross value). For the purpose of this section, an index TL (test load) is used either for emply or filled containers.

The procedure could be based either on OIML R 111 [3] or EURAMET Calibration Guide No. 18. The section 4.4.1 deals with the case where the control instrument is used as a mass comparator and the conventional mass of the test load is obtained by its calibration with a reference standard weight. The section 4.4.2 deals with the case where the test load is weighed on the calibrated control instrument.

These two approaches for determining the conventional mass of the test fills are further discussed in the section 4.6 in order to determine the reference value of mass of test fills.

4.4.1 Determination of the conventional mass of the test load based on OIML R111

If the procedure for determination of the conventional mass and uncertainty of the test load is based on OIML R111, the control instrument is used as a mass comparator and the conventional mass of the test load m_{cTL} is obtained by its calibration with a reference standard weight. In general, the relation is the following:

$$m_{cTL} = m_{cR}(1 + C) + \overline{\Delta m_c} \tag{4.4.1-1}$$

where

$$C = (\rho_{aCI} - \rho_0)\left(\frac{1}{\rho_{TL}} - \frac{1}{\rho_R}\right) \tag{4.4.1-2}$$

with

m_{cR} – conventional mass of the standard weights used for calibration of the test load on the control instrument,

C – air bouynacy correction contribution

$\overline{\Delta m_c}$ – average measured difference between the test load and standard weights

ρ_{aCI} - air density at the time of calibration of the test load on the control instrument,

ρ_0 - reference air density, 1,2 kg/m^3,

ρ_{TL} - density of the test load,

ρ_R - density of standard weights used for calibration of the test load on the control instrument,

4.4.2 Determination of the conventional mass of the test load based on EURAMET Calibration Guide No. 18

If the procedure the procedure for determination of the conventional mass and uncertainty of the test load is based on EURAMET Calibration Guide No. 18, the test load is weighed on the calibrated control instrument. The conventional mass of the test load m_{cTL} is proportional to the weighing result of the control instrument W_{CI}. The relation is the following [2]:

$$m_{cTL} = W_{CI}\left[1 + \left(\rho_{aCI} - \rho_0\right)\left(\frac{1}{\rho_{TL}} - \frac{1}{\rho_{sCI}}\right)\right] \tag{4.4.2-1}$$

where

$$W_{CI} = R_{CI} - E_{CI} \qquad (4.4.2\text{-}2)$$

$$R_{CI} = R_{LCI} - R_{0CI} \qquad (4.4.2\text{-}3)$$

$$E_{CI} = I_{CI} - m_{refCI} \qquad (4.4.2\text{-}4)$$

$$I_{CI} = I_{LCI} - I_{0CI} \qquad (4.4.2\text{-}5)$$

$$m_{refCI} = m_{cCalCI} + \delta m_{BCI} \qquad (4.4.2\text{-}6)$$

$$\delta m_{BCI} = -m_{cCalCI}\left[(\rho_{aCalCI} - \rho_0)\left(\frac{1}{\rho_{CalCI}} - \frac{1}{\rho_{sCI}}\right)\right] \qquad (4.4.2\text{-}7)$$

with
R_{CI} – reading of the test load on the control instrument corrected for zero reading
R_{LCI} – reading of the test load on the control instrument (loaded)
R_{0CI} - reading of the test load on the control instrument (unloaded)
E_{CI} – eror of the control instrument
I_{CI} – indication of the standard weights on the control instrument corrected for zero indication
I_{LCI} – indication of the standard weights on the control instrument (loaded)
I_{0CI} - indication of the standard weights on the control instrument (unloaded)
m_{refCI} – reference value of mass of standard weights used for calibration of the control instrument,
m_{cCalCI} - conventional mass of the standard weights used for calibration of the control instrument,
δm_{BCI} – air bouyancy correction for the standard weights used for calibration of the control instrument,
ρ_{aCI} - air density at the time of weighing of the test load on the control instrument,
ρ_{aCalCI} - air density at the time of calibration of the control instrument with mass,
ρ_{sCI} - density of standard weights used for adjustment of the control instrument,
ρ_{CalCI} - density of standard weights used for calibration of the control instrument,
ρ_{TL} - density of the test load,
ρ_0 - reference air density, 1,2 kg/m^3,

4.4.3 Standard weights

Standard OIML weights directly applied on the instrument are not used for calibration of the instrument.

The requirements for the standard weights, which are used for calibration of the control instrument or calibration of the test loads using the control instrument as a comparator are given in [2] and [3], respectively.

4.4.4 Effects of convection

The test fills may not be at the same temperature as the surrounding air, but in general the effect of convenction may be neglected.

At extreme initial temeprature difference ΔT_0 between the test fills and surrounding air the effect of convection may be reduced to a smaller value ΔT over a time Δt allowing the test fills to reach the temperature of the environment before determing their conventional mass on the control instrument, i.e. to the extent that the remaining change Δm_{conv} is negligible in view of the uncertainty of the calibration required by the client. In such a case, it is practically not possible to perform the correction for the convenction effects, therefore the error due to convection intrisically forms part of the preset value error.

4.5.5 *Matterial loss*

To be added.

4.5 Indications

4.5.1 General

An automatic weighing instrument shall be capable of performing consecutive weighing cycles without any intervention of an operator [4]. Unlike for non-automatic weighing instrument, the automatic weighing instrument operator is niether capable nor expected to make a correction of the indication under load with the indication at no load.

Furthemore, for the purpose of calibration of automatic gravimetric filling instrument, only the preset value of fill F_P is taken into account,

During calibration tests, the indications of calibrated intrument, if available at all, are not recorded.

4.5.2 Resolution

The preset value of fill is introduced as integer multiples of the scale interval *d*. This value is considered as a resolution of the instrument.

4.6 Reference value of mass of test fills

To determine the preset value error of an instrument E_P, the reference values of mass of test fills $m_{ref,i}$ need to be known. The conventional masses of test fills is a priori not known, density of filled product ρ_F is normally significantly different from the reference value ρ_c and the air density ρ_a at the time of calibration is normally different from ρ_0.

The preset value error for i-th test fill equals:

$$E_{P,i} = m_{ref,i} - F_P \tag{4.6-1}$$

Due to effects of air buoyancy and others, which may lead to minor correction terms δm_x, $m_{ref,i}$ is not exactly equal to $m_{cF,i}$, the conventional mass of i-th test fill

$$m_{ref,i} = m_{cF,i} + \delta m_{B,F} + \delta m \ldots \tag{4.6-2}$$

with

$\delta m_{B,F}$ – air bouyancy correction for the test fill (refers to net value of test fill),

δm — further corrections that it may be necessary to apply under special conditions, e.g. correction due to possible drift of the test load or due to convection effects, are not considered hereafter.

The conventional mass of test fill cannot be determined directly, but trough determination conventional mass of the filled container m_{cB} (gross value) and conventional mass of the empty container m_{cT} (tare value)

$$m_{cF,i} = m_{cB,i} - m_{cT,i} \tag{4.6-3}$$

and

$$m_{ref,i} = m_{cB,i} - m_{cT,i} + \delta m_{B,F} + \delta m \ldots \tag{4.6-4}$$

The air bouyancy correction for the test fill $\delta m_{B,F}$ is affected by air density at the time of calibration of the instrument ρ_a, density of the test fill ρ_F and density of standard weights used for adjustment of the AGFI ρ_s

$$\delta m_{B,F} = -m_{c,F}\left[(\rho_a - \rho_0)\left(\frac{1}{\rho_F} - \frac{1}{\rho_s}\right)\right] \tag{4.6-5}$$

with reference air density $\rho_0 = 1{,}2\ kg/m^3$.

In (4.6-5), $m_{c,F}$ can be replaced, without introducing a significant error, with nominal mass of the test fill $m_{N,F}$, which equals the preset value of fill F_P

$$m_{N,F} = F_p \tag{4.6-6}$$

Opposite to calibration of NAWI, density of the test load used at calibration of the AGFI, i.e. the filled product, may significantly differ from reference density of standard weights ρ_s, i.e. 8000 kg/m^3 [5]. However, the conventional mass of test fills is determined at the time and place of calibration of the AGFI and at the same air density, and it will be shown that the density of the filled product and the density of the containers don't need to be known.

m_{ref} could be determined in various ways as it is summarised in the table below.

Section	Principle of calibration of test load	Time of calibration	Remarks
4.6.x	Test fills calibrated on the control instrument used as comparator (calibration based on OIML R111)	Test fills calibrated at the time and place of calibration of AWI	
4.6.1	Test fills weighed on the calibrated control instrument	Control instrument calibrated at time and place of calibration of AWI Test load weighed at the time and place of calibration of AWI	
4.6.2	Test fills weighed on the calibrated control instrument	Control instrument calibrated previously. Test load weighed at the time and place of calibration of AWI	Control instrument needs to have calibration certificate. Uncertainty of control instrument in use needs to be taken into account.
4.6.3	Test fills weighed on the verified control instrument	Control instrument verified previously. Test load weighed at the time of calibration of AWI.	Traceability of the verification needs to be demonstrated. Uncertainty of control instrument in use needs to be taken into account.

Sections 4.6.1 to 4.6.3 provide examples of determination of reference value of mass of test fill $m_{ref.i}$ based on determination of the conventional mass of test fill $m_{cF,i}$ (trough determination $m_{cB,i}$ and $m_{cT,i}$) and the air buoyancy correction for test fill $\delta m_{B,F}$, considering the principle and time of calibration of the test fills

$$m_{ref,i} = m_{cB,i} - m_{cT,i} + \delta m_{B,F}. \tag{4.6-7}$$

Further in this section, index i is not used anymore, however equations refere to i -th test fill.

4.6.1 Test fills weighed on the control instrument calibrated at time and place of calibration of the AGFI. Test fills weighed at the time and place of calibration of the AGFI.

The control instrument should be calibrated in calibration points close to the preset values of fills and the error of indication of the control instrument is taken into account.

Based on (4.4.2-1) to (4.4.2-7) for the conventional mass of the test load, and under condition that $(R_{LCI}-R_{0CI})-(I_{LCI}-I_{0CI})-m_{cCalCI}\left[(\rho_{aCalCI}-\rho_0)\left(\frac{1}{\rho_{CalCI}}-\frac{1}{\rho_{sCI}}\right)\right] \ll m_{cCalCI}$ and $m_{cTL} \approx m_{cCalCI}$, we get the following general expression for for m_{cB} and m_{cT}

$$m_{cB} = \left(R_{LCI,B}-R_{0CI,B}\right)-\left(I_{LCI,B}-I_{0CI,B}\right)+m_{cCalCI,B}-\left(m_{N,F}+m_{N,T}\right)\left[(\rho_{aCalCI}-\rho_0)\left(\frac{1}{\rho_{CalCI}}-\frac{1}{\rho_{sCI}}\right)\right]+\left(m_{N,F}+m_{N,T}\right)(\rho_{aCI}-\rho_0)\left(\frac{1}{\rho_F}-\frac{1}{\rho_{sCI}}\right) \quad (4.6.1\text{-}1)$$

where

$$m_{cCalCI,B} \approx m_{N,F}+m_{N,T} \quad (4.6.1\text{-}2)$$

and

$$m_{cT} = \left(R_{LCI,T}-R_{0CI,T}\right)-\left(I_{LCI,T}-I_{0CI,T}\right)+m_{cCalCI,T}-m_{N,T}\left[(\rho_{aCalCI}-\rho_0)\left(\frac{1}{\rho_{CalCI}}-\frac{1}{\rho_{sCI}}\right)\right]+m_{N,T}(\rho_{aCI}-\rho_0)\left(\frac{1}{\rho_T}-\frac{1}{\rho_{sCI}}\right) \quad (4.6.1\text{-}3)$$

Based on (4.6-7) for the reference value of mass of test fill, (4.6.1-1) and (4.6.1-3) for m_{cB} and m_{cT}, respectively, and (4.6-5) for the air bouyancy correction, and taking into account that the reference value of mass of test fills is determined at the same time and place as calibration of the AGFI, then $\rho_{aCI}=\rho_{aCalCI}=\rho_a$, we get the following general expression for m_{ref}

$$m_{ref} = \left(\left(R_{LCI,B}-R_{0CI,B}\right)-\left(I_{LCI,B}-I_{0CI,B}\right)+m_{cCalCI,B}\right)-\left(\left(R_{LCI,T}-R_{0CI,T}\right)-\left(I_{LCI,T}-I_{0CI,T}\right)+m_{cCalCI,T}\right)-m_{n,F}(\rho_a-\rho_0)\left(\frac{1}{\rho_{CalCI}}-\frac{1}{\rho_s}\right) \quad (4.6.1\text{-}4)$$

The total contribution of the air buoyancy correction δm_{BTot} to m_{ref} in (4.6.1-4) equals

$$\delta m_{BTot} = -m_{n,F}(\rho_a-\rho_0)\left(\frac{1}{\rho_{CalCI}}-\frac{1}{\rho_s}\right) \quad (4.6.1\text{-}5)$$

and

$$m_{ref} = W_B' - W_T' + \delta m_{BTot} \quad (4.6.1\text{-}6)$$

where

$$W_B' = \left(R_{LCI,B}-R_{0CI,B}\right)-\left(I_{LCI,B}-I_{0CI,B}\right)+m_{cCalCI,B} \quad (4.6.1\text{-}7)$$
$$W_T' = \left(R_{LCI,T}-R_{0CI,T}\right)-\left(I_{LCI,T}-I_{0CI,T}\right)+m_{cCalCI,T} \quad (4.6.1\text{-}8)$$

4.6.2 Test fills weighed on the previously calibrated control instrument. Test fills weighed at the time and place of calibration of the AGFI

The reference value of mass of test fills is determined by weighing of filled and empty containers on the control instrument, which has been calibrated previously. The calibration certificate for the control instrument is on a disposal. However, the same approach could be used also in a case when the instrument is calibrated immediately prior to determination of the conventional mass of test fills.

If the gross value (filled containers) and tare value (empty containers) of test fills are close to the calibration points in which the errors of control instrument were determined, then the weighing result W_{CI} (either for the gross or for tare value) could be determined based on the reading of the filled or empty containers mass values R_{CI} corrected for the error of the control instrument E_{CI} as given by (4.4.2-2). The error of the control instrument is reported in its calibration certificate.

$$W_{CI} = R_{CI} - E_{CI}$$

If this is not the case (e.g. when the conventional mass of filled or empty containers is not close to the calibration point in which the error of control instrument was determined, or if so decided by the calibration laboratory), no correction is applied to the reading

$$W_{CI} = R_{CI} \tag{4.6.2-1}$$

but errors of the control instrument need to be included in an uncertainty (i.e. a "global uncertainty" $U_{gl}(W_{CI})$, which includes the errors of indication such that no corrections have to be applied to the readings in use).

For a reading taken under the same conditions as those prevailing at calibration of the control instrument (e.g. immediatelly after its adjustment), the result may be denominated as the weighing result under conditions of the calibration W_{CI}^{*}.

Based on (4.4.2-1) for the conventional mass of the test load we get the following expressions for m_{cB} and m_{cT}

$$m_{cB} = W_{CI,B} + \left(m_{N,F} + m_{N,T}\right)(\rho_{aCI} - \rho_0)\left(\frac{1}{\rho_F} - \frac{1}{\rho_{sCI}}\right) \tag{4.6.2-2}$$

where

$$W_{CI,B} \approx m_{N,F} + m_{N,T} \tag{4.6.2-3}$$

and

$$m_{cT} = W_{CI,T} + m_{N,T}(\rho_{aCI} - \rho_0)\left(\frac{1}{\rho_T} - \frac{1}{\rho_{sCI}}\right) \tag{4.6.2-4}$$

with
$W_{CI,B}$ – weighing result for filled container (gross),
$W_{CI,T}$ - weighing result for empty container (tare),
$m_{N,F}$ – nominal mass of the test fill,
$m_{N,T}$ – nominal mass of the empty container,
ρ_F – density of the filled product,
ρ_T – density of the container.

Based on (4.6-7) for the reference value of mass of test fill, (4.6.2-2) and (4.6.3-4) for m_{cB} and m_{cT}, respectively, and (4.6-5) for the air bouyancy correction, and taking into account that the reference value of mass of test fills is determined at the same time and place as calibration of the AGFI, $\rho_{aCI} = \rho_a$, we get the following general expression for m_{ref}

$$m_{ref} = W_{CI,B} - W_{CI,T} - m_{n,F}(\rho_a - \rho_0)\left(\frac{1}{\rho_{sCI}} - \frac{1}{\rho_s}\right) \tag{4.6.2-5}$$

The total contribution of the air buoyancy correction δm_{BTot} to m_{ref} in (4.6.3-5) equals

$$\delta m_{BTot} = -m_{n,F}(\rho_a - \rho_0)\left(\frac{1}{\rho_{sCI}} - \frac{1}{\rho_s}\right) \tag{4.6.2-6}$$

and

$$m_{ref} = W_{CI,B} - W_{CI,T} + \delta m_{BTot} \tag{4.6.2-7}$$

4.6.3 Test fills weighed on the previously verified control instrument. Test fills weighed at the time and place of calibration of the AGFI

The reference value of mass of test fills is determined by weighing of filled and empty containers on the control instrument, which has been verified. Traceability of the verification needs to be demonstrated.

In general, no correction is applied to the reading (as in (4.6.2-1))

$$W_{CI} = R_{CI}$$

but errors of the verified control instrument need to be included in an uncertainty (i.e. a “global uncertainty” $U_{gl}(W_{CI})$, which includes the errors of indication such that no corrections have to be applied to the readings in use).

Since the reference value of mass of test fills is determined at the same time and place as calibration of the AGFI, $\rho_{aCI} = \rho_a$, m_{ref} and δm_{BTot} are determined in the same way as in the section 4.6.2, i.e. using (4.6.2-7) and (4.6.2-6).

4.7 Operating parameters

Type of the test material, rate of operation must be recorded for each case [1].

5 MEASUREMENT METHODS

Tests are normally performed to determine the preset value error at different preset values.

The calibration laboratory deciding on the number of measurements for its routine calibration procedure should consider that, in general, a larger number of measurements tends to reduce the uncertainty of measurement but increase the cost.

Details of the tests performed for an individual calibration may be fixed by agreement of the client and the calibration laboratory, in view of the normal use of the instrument. The parties may also agree on further tests or checks, which may assist in evaluating the performance of the instrument under special conditions of use. Any such agreement should be consistent with the minimum numbers of tests as specified in the following sections.

The general test procedure for the test specified in the following sections shall be as follows:

1) Select the test fills material as specified in the section 4.3 and the preset values of fills as specified in the section 5.1.

2) Determine the conventional mass of empty container(s) on the control instrument as specified in the sections 5.2 and 6.1.

3) The instrument should be tested in its normal mode of automatic operation. Start the automatic weighing system, including the surrounding equipment, which is normally operational when the instrument is in use.

4) Set the preset value of fills and set the rate of operation agreed with the client. Normally this is the rate of operation used by the client. This may vary depending on the preset value.

5) The number of consecutive test fills depends on the preset value as specified in the section 5.1.

6) Zero shall be set at the start of each test at a given load value and not readjusted at any time during the test. It has to be allowed that automatic adjustment of zero is operational during the tests.

7) Enable automatical filling of the specified number of test fills.

8) Determine the conventional mass of each test fill on the control instrument as specified in the section 6.1.

5.1 Test for preset value error and repeatability

The test consists of repeated filling the sfills with the same nominal value, and under constant test conditions. The purpose of this test is an evaluation of the accuracy and repeatability of the instrument at least in some points of usual application of the instrument if not over the whole weighing range. Each test point is characterized by its own repeatability.

The test is performed with k different preset values F_{Pj} , $1 \leq j \leq k$.

Examples for the preset value target values:

- the minimum nominal value of fills produced on-site *Min'*,
- the largest nominal value of fills produced on-site *Max'*,
- a value equally distributed between *Min'* and *Max'* according, especially if *Min'* is less than one third of *Max'*.

Details about the target preset values need to be agreed with the client to represent usual use of the calibrated instrument.

The minimum number of individual test fills shall be as specified in the following table:

Preset value of the fills, F_p	Minimum number of test fills, n
$F_p \leq 1$ kg	60
1 kg < $F_p \leq 10$ kg	30
10 kg < $F_p \leq 25$ kg	20
25 kg < F_p	10

5.2 Containers tare value

The tare of empty containers, i.e. their conventional mass needs to be determined on the control instrument as a part of the calibration.

Case A:
If the mass difference between individual empty containers is equal or larger then 0,5 d (a half of the scale interval value of preset value device) of the calibrated AGFI, then it is necessary to determine the conventional mass of each empty container, which will be used.

Case B:
If the mass of containers is virtually constant, i.e. the mass difference between individual containers is smaller then 0,5 d of the scale interval value of preset value device, it is sufficient to determine avegarage conventional mass of empty containers.

The average conventional mass of empty containers could be determined by simultaneous weighing of a sample of several empty containers (e.g. 10 or more).

5.3 Auxiliary measurements

The air temperature in reasonable vicinity to the instrument should be measured, at least once during the calibration.

If it is necessary to prevent excessive convection effects, a limiting value for the temperature difference between the test loads and instrument should be observed, and/or an acclimatisation time that has been executed recorded.

6 MEASUREMENT RESULTS

The procedures and formulae in the chapters 6 and 7 provide the basis for the evaluation of the results of the calibration tests and therefore require no further description on a calibration certificate. If the procedures and formulae used deviate from those given in the guide, additional information may need to be provided in the certificate.

It is not intended that all of the formulae, symbols and/or indices are used for presentation of the results in the calibration certificate.

6.1 Preset value error

For j-th preset value F_{Pj}, the preset value error E_{Pj} is calculated based on measurement of n test fills as follows

$$E_{Pj} = \frac{1}{n}\sum_{i=1}^{n} m_{ref,ij} - F_{Pj} \tag{6.1-1}$$

When the test fills are weighed on the previously calibrated or verified control instrument (cf. sections 4.6.2 and 4.6.3, respectively), (6.1-1) can be modified by introduction of weighing results for filled and empty containers in the equation.

If according to Section 5.2 it is necessary to measure each empty container (Case A), (6.1-1) is modified, taking into account (4.6.2-7), to

$$E_{Pj} = \frac{1}{n}\sum_{i=1}^{n}\left(W_{CI,Bij} - W_{CI,Tij}\right) + \delta m_{BTot,j} - F_{Pj} \tag{6.1-2a}$$

If according to Section 5.2 it is suitable only to measure a sample of several empty containers (Case B), (6.1-1) is modified, taking into account (4.6.2-7), to

$$E_{Pj} = \frac{1}{n}\sum_{i=1}^{n}\left(W_{CI,Bij} - \frac{1}{m}\sum_{z=1}^{m} W_{CI,Tzj}\right) + \delta m_{BTot,j} - F_{Pj} \tag{6.1-2b}$$

From (6.1-2a), mass of a single fill F_{ij} is defined as

$$F_{ij} = W_{CI,Bij} - W_{CI,Tij} \tag{6.1-3a}$$

and from (6.1-2b) as

$$F_{ij} = W_{CI,Bij} - \frac{1}{m}\sum_{z=1}^{m} W_{CI,Tzj} \tag{6.1-3b}$$

For the case when the test fills are weighed on the control instrument calibrated at time and place of calibration of the AGFI (cf. sections 4.6.1), W_B and W_T in (6.1-2a) to (6.1-3b) are replaced by W'_B and W'_T respectively, as given in (4.6.1-7) and (4.6.1-8)

Finally

$$E_{Pj} = \overline{F}_j + \delta m_{BTot} - F_{Pj} \tag{6.1-4}$$

where $\overline{F}_j$ is average mass of fills

$$\overline{F}_j = \frac{1}{n}\sum_{i=1}^{n} F_{ij} \tag{6.1-5}$$

6.2 *Repeatability*

From the n fills F_{ij}, $i = 1, \ldots, n$, for a given j-th preset value F_{Pj}, the standard deviation $s(F_j)$ is calculated

$$s(F_j) = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}\left(F_{ij} - \overline{F}_j\right)^2} \tag{6.2-1}$$

7 UNCERTAINTY OF MEASUREMENT

In this and the following sections, there are uncertainty terms assigned to small corrections. For the quotient of such an uncertainty divided by the related value of mass or indication, the abbreviated notation u_{rel} will be used.

Example: let

$$u(\delta m_{corr}) = m \cdot u(corr) \tag{7-1}$$

with the dimensionless term $u(corr)$, then

$$u_{rel}(\delta m_{corr}) = u(corr) \tag{7-2}$$

Accordingly, the related variance will be denoted by $u_{rel}^2(\delta m_{corr})$ and the related expanded uncertainty by $U_{rel}(\delta m_{corr})$.

For the determination of uncertainty, second order terms have been considered negligible, but when first order contributions cancel out, second order contributions should be taken into account (see JCGM 101 [6], 9.3.2.6).

7.1 *Standard uncertainty of the preset value error*

The basic formula for the calibration is (the same as (6.1-1), only the index j is omitted for the sake of simplicity))

$$E_P = \frac{1}{n}\sum_{i=1}^{n} m_{ref,ij} - F_P \tag{7.1-1}$$

To account for sources of variability of the preset value error, (7.1-1) is amended by correction terms δX_{xx} as follows

$$E_P = \frac{1}{n}\sum_{i=1}^{n} F_i + \delta F_{rep} + \delta F_{repT} + \delta m_{BTot} - F_P + \delta I_{dig} \tag{7.1-2}$$

or

$$E_P = \frac{1}{n}\sum_{i=1}^{n}(W_{Bi} - W_{Ti}) + \delta F_{rep} + \delta F_{repT} + \delta m_{BTot} - F_P + \delta I_{dig} \tag{7.1-3}$$

with standard uncertainty of the preset error

$$u(E_P) = \sqrt{u^2(W_B) + u^2(W_T) + u^2(F_{rep}) + u^2(F_{repT})^2 + u^2(m_{BTot}) + u^2(\delta I_{dig})} \quad (7.1\text{-}4)$$

All input quantities are considered to be uncorrelated, therefore covariances are not considered.

The terms are further expanded hereafter.

7.1.1 Standard uncertainty of the weighing result for filled containers (gross)

Taken into account the content of table in Section 4.6, the following cases are treated in this section:

A. Test load weighed on the simultaneoulsy calibrated control instrument
B. Test load weighed on the previously calibrated control instrument
C. Test load weighed on the previously verified control instrument

In general, the uncertainty of weighing result gets larger from procedure A to procedure C.

7.1.1.A Filled container weighed on the simultaneulsy calibrated control instrument

To account for sources of variability of the weighing result, when a control instrument is used, which was calibrated immediately before the weighing took place, (4.6.1-7) is amended by corrections terms δX_{xx} as follows

(7.1.1-1)

$$\begin{aligned} W_B' = &\left(R_{LCI,B} + \delta R_{digLCI,B} + \delta R_{repCI,B} + \delta R_{eccCI,B} - \left(R_{0CI,B} + \delta R_{0CI,B}\right)\right) \\ &- \left(I_{LCI,B} + \delta I_{digLCI,B} + \delta I_{repCI,B} + \delta I_{eccCI,B} - \left(I_{0CI,B} + \delta I_{dig0CI,B}\right)\right) \\ &+ \left(m_{NCalCI,B} + \delta m_{cCalCI,B} + \delta m_{DCalCI,B} + \delta m_{convCalCI,B}\right) \end{aligned}$$

7.1.1.1 $R_{CI,B}$ is the reading of the filled container on the control instrument. Its standard uncertainty $u(R_{CI,B})$ should be evaluated according to Section 7.4.1 of cg-18 [2].

7.1.1.2 $I_{CI,B}$ is indication of the standard weights on the control instrument. Its standard uncertainty $u(I_{CI,B})$ should be evaluated according to Section 7.1.1 of cg-18 [2].

7.1.1.3 $m_{CalCI,B}$ is the reference mass value of standard weights used for calibration of the control instrument at gross value fo the test fills, without taking into account the correction term for air buoyancy. Its standard uncertainty $u(m_{CalCI,B})$ should be evaluated according to Section 7.1.2 of cg-18 [2], but not taking into account the standard uncertainty of the air bouyancy correction. The standard uncertainty of the air bouyancy correction is treated separately according the section 7.1.5.

7.1.1.4 When the filled containers are weighed on the simultaneulsy calibrated control instrument, the standard uncertainty of the reference mass is obtained from

$$u^2(W_B) = u^2(R_{CI,B}) + u^2(I_{CI,B}) + u^2(m_{CalCI,B}) \quad (7.1.1\text{-}2)$$

with the contributions from 7.1.1.1 to 7.1.1.3.

7.1.1.B Filled container weighed on the previously calibrated control instrument

7.1.1.4 When a previously calibrated control instrument is used, W_B is determined according to the section 4.6.2. Its standard uncertainty $u(W_B)$ should be evaluated according to the sections 7.4.5 or 7.5.2 of cg-18 [2] for a case when errors of the control instrument are accounted by correction or included in a "global" uncertainty $U_{gl}(W_{CI})$, respectively.

According to cg-18 [2], standard uncertainty for the weighing result under conditions of the calibration $u(W_{CI}^{*})$ could be used instead of $u(W_{CI})$ if the control instrument was calibrated right before its use. Similar can be assumed if the control instrument was adjusted right before its use and uncertainty contributions resulting from the opration of the control instrument (as defined in Section 7.4.4 of cg-18 [2]) are negligable.

The control instruments needs to have the calibration certificate. If the certificate states uncertainty of the instrument in use, this is non-acredited value. The laboratory needs to independently evaluate uncertainty in use based on actual conditions valid in a period since the last calibration of control instrument.

7.1.1.C Test load weighed on the previously verified control instrument

7.1.1.5 When a previously verified control instrument is used, W_{CI} is determined according to the section 4.6.3. Its standard uncertainty $u(W_{CI})$ should be evaluated according to the section 7.5.2 of cg-18 [2] for a case when errors of the control instrument are included in a “global” uncertainty $U_{gl}(W)$. In general, errors of the control instrument are not know, but durinq the verificiation they were smaller or equal to the maximum permissible error of the weighing instrument mpe_{R76}[7,8]. It can be assumed that errors of the control instrument are evenly distributed in the range defined by mpe_{R76}.

The stadard global uncertainty of the verified control instrument $u_{gl}(W_{CI})$ can be estimated by:

$$u_{gl}(W_B) = \sqrt{mpe_{R76}{}^2 + \left(\frac{mpe_{R76}}{\sqrt{3}}\right)^2} \cong 1{,}15 mpe_{R76} \qquad (7.1.1\text{-}2)$$

7.1.2 Standard uncertainty of the weighing result for empty container (tare)

For evaluation of standard uncertainty of the weighing result for empty container $u(W_T)$, a relevant approach from the section 7.1.1 need to be used, which is in agreement with an applied principle of determination of the weighing result of empy containers from the section 4.6, and applied to W_T instead of W_B.

If the average conventional mass of containers is determined by simultaneous weighing of a sample of several empty containers, uncertainty is in the first step estimated for the complete sample $u(W_{mT})$, then, in the second step, uncertainty of an individual empty container equals

$$u(W_T) = \frac{1}{m} u(W_{mT}) \qquad (7.1.2\text{-}1)$$

7.1.3 Standard uncertainty of the mean of several fills

δF_{rep} accounts for the repeatability of test fills weighed on the control instrument; normal distribution is assumed, estimated as

$$u(\delta F_{rep}) = s(F_j)/\sqrt{n} \qquad (7.1.3\text{-}1)$$

where $s(F_j)$ is determined in 6.2 and n is number of repeated fills.

$s(F_j)$ is determined for each given preset value F_P and it is considered as representative only for the respective value.

7.1.4 Standard uncertainty of variability of tare

δF_{repT} accounts for the variability of mass of empty containers. If the mass of containers is virtually constant (cf. Case B in 5.2), i.e. the mass difference between individual containers is smaller then 0,5 d of the scale interval value of preset value device; rectangular distribution is assumed, estimated as

$$u(\delta F_{repT}) = \frac{d/2\sqrt{3}}{m} \quad (7.1.4\text{-}1)$$

where m is number of empty containers simultaneously weighed on the control instrument.

δF_{repT} is determined for each given type of container used druging the calibration.

This contribution is not applicable in the case, where tare value of each container is determined and taken into account (cf. Case A in 5.2).

7.1.5 Standard uncertainty of air buoyancy correction

δm_{BTot} is the correction for air buoyancy as introduced in (4.6.1-5) and (4.6.2-6).

Where a simple comparison of expected values of δm_{BTot} with the resolution of the preset value device d shows that the air buoyancy correction is small enough, a more elaborate calculation of the correction and its uncertainty component based on actual data may be superfluous. Consequently, no correction is applied, i.e. δm_{BTot}=0. Where conformity of the standard weights used for adjustment of the AGFI and control instrument to OIML R111 [3] is established and if the AGFI is not adjusted before calibration, the relative uncertainty is evaluated as

$$u_{rel}(\delta m_{BTot}) \approx (0{,}1\,\rho_0/\rho_c + mpe/(4m_N))/\sqrt{3} \quad (7.1.5\text{-}1)$$

For smaller uncertainties, e.g. if air buoyancy correction is taken into account according to (4.5.4-5) or AGFI is adjusted immediately before calibration, the standard uncertainties could be evaluated according Section 7.1.2.1 of cg-18 [2].

7.1.6 Standard uncertainty of the rounding error of preset value

δI_{dig} accounts for the rounding error of preset value. Its expectation value of correction is zero. Limits of δI_{dig} are $\pm d/2$; rectangular distribution to be assumed, therefore it's standard uncertainty equals

$$u(\delta I_{dig}) = d/2\sqrt{3} \quad (7.1.6\text{-}1)$$

7.2 Expanded uncertainty at calibration

The expanded uncertainty of the preset value error is

$$U(E_p) = ku(E_p) \quad (7.2\text{-}1)$$

The coverage factor $k = 2$ should be chosen such that the expanded uncertainty corresponds to a coverage probability of 95,45 %.

8 REFERENCES

[1] *OIML R 61-1: Automatic gravimetric filling instruments. Part 1: Metrological and technical requirements – Tests*, Edition 2004 (E)

[2] *EURAMET Calibration Guide No. 18: Guidelines on the Calibration of Non-Automatic Weighing Instruments*, Version 4.0 (1/2015)

[3] *OIML R111, Weights of Classes E1, E2, F1, F2, M1, M1-2, M2, M2-3, M3,* Edition 2004 (E)

[4] *Welmec 2, Directive 2009/23/EC: Common application non-automatic weighing instruments*, 2015

[5] *OIML D 28: Conventional value of the result of weighing in air*, Edition 2004 (E)

[6] *JCGM 101:2008, Evaluation of Measurement Data – Supplement 1 to the "Guide to the expression of uncertainty in measurement" – Propagation of Distributions using a Monte Carlo method,* 1st edition, 2008

[7] *OIML R 761-1: Non-automatic weighing instruments. Part 1: Metrological and technical requirements – Tests*, Edition 2006 (E)

[8] *EN 45501:2015 Metrological aspects of non-automatic weighing instruments*