White Paper

Calibration of Weighing Instruments

Weighing in the Safe Weighing Range

Contents

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1. Calibration, Adjustment and Verification

Calibration is one of the key activities that must be performed periodically when instruments are used for quality relevant measurements. Internationally, there are many standards which stipulate this requirement, e.g. ISO9001, GMP regulations or standards concerned with food safety. Unfortunately, there is no common understanding on the definition, the implementation and the specific activities that comprise calibration. Let us therefore start by establishing a common platform on what calibration is.

Calibration is a set of activities carried out on a measurement instrument to understand its behavior by establishing a relationship between known values (measurement standards) and the associated measured values (indications). The relationship consists of a deviation and its associated uncertainty. The "International Vocabulary of Metrology" (VIM)[1] provides the official definition of calibration:

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

It is evident that the relation between the known and the measured values can only be established if the associated measurement uncertainties are derived. Basically, measurement uncertainty describes how far away from the true value a measurement result reasonably might be.

Besides calibrating, an instrument can also be adjusted. Adjustment is defined in the "International Vocabulary of Metrology" (VIM) as follows:

"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."

In other words, when adjusting an instrument, its indications are modified in a way so that they correspond – as far as possible – to the quantity values of the measurement standards applied. Unfortunately, many users apply the words calibration and adjustment interchangeably, incorrectly or even randomly. Quite often, they talk about calibrating a weighing instrument, however they mean adjusting it. The VIM also emphasizes this by stating:

"Adjustment of a measuring system should not be confused with calibration, which is a prerequisite for adjustment. After an adjustment of a measuring system, the measuring system must usually be recalibrated."

This statement highlights another important aspect of calibration: Before an instrument is adjusted, it must be first calibrated in order to understand – and document – its behavior. Equally after an adjustment, the instrument must usually be recalibrated. Quite often, users talk about an "as found" calibration, i.e. a calibration of the instrument before any modification (adjustment) is carried out, and about an "as left" calibration, i.e. a calibration of the instrument after any necessary adjustment and/or repair has been carried out.

Besides of calibration, measuring instruments can also be verified. Usually, instruments need to fulfill predefined requirements, quite frequently expressed as tolerances. The "International Vocabulary of Metrology" (VIM) defines verification as follows:

"Provision of objective evidence that a given item fulfil specified requirements."

While calibration only establishes the relationship between measurement standards and indications ("how well performs the instrument"), verification assesses the instrument on whether or not it meets specific requirements ("does the instrument perform well enough"). Usually, the outcome of verification is a "pass" or a "fail". In respect to weighing instruments, the requirements can come from the manufacturer who specifies tolerances for each balance or scale model, international or national testing recommendations and handbooks for weighing instruments used for applications involving commercial transactions (like OIML R76-1[2] or HB44[3]) as well as industry specific regulations (like USP General Chapter 41[4]). However, even more importantly, the user needs to specify weighing tolerances that assure that the instrument performs well enough to fulfill his specific process requirements. In view of the application of the weighing instruments, these tolerances are the most important ones as they have a direct impact on the quality of the final product.

2. Calibration Procedure of EURAMET cg-18

In an effort to harmonize the requirements for the calibration of non-automatic weighing instruments (NAWI) on an international level, the guideline EURAMET cg-18 "Guidelines on the calibration of non-automatic weighing instruments" was developed by the leading European metrology institutes and is now widely applied by calibration laboratories in Europe[5]. The guide has been adopted by SIM (Sistema Interamericano de Metrología)[6] and thus is formally recognized by the regional American metrology organizations. There are recent activities in the US, triggered by ASTM, who are interested in taking over the methodology of cg-18 and transposing it into an ASTM standard, which could potentially serve as a future national calibration guide for the US.

The first step in calibration is deriving the so-called standard uncertainty $u(E)$ for the error of indication E of the selected calibration points. The basic formula for that step defines the error of indication as the deviation of the indication from the reference value of mass

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

and derives the associated standard uncertainty as

$$u^2(E) = \frac{d_0^2}{12} + \frac{d_L^2}{12} + s^2(I) + u_{rel}^2(\delta I_{ecc})I^2 + u^2(\delta m_c) + u^2(\delta m_B) + u^2(\delta m_D) + u^2(\delta m_{conv})$$

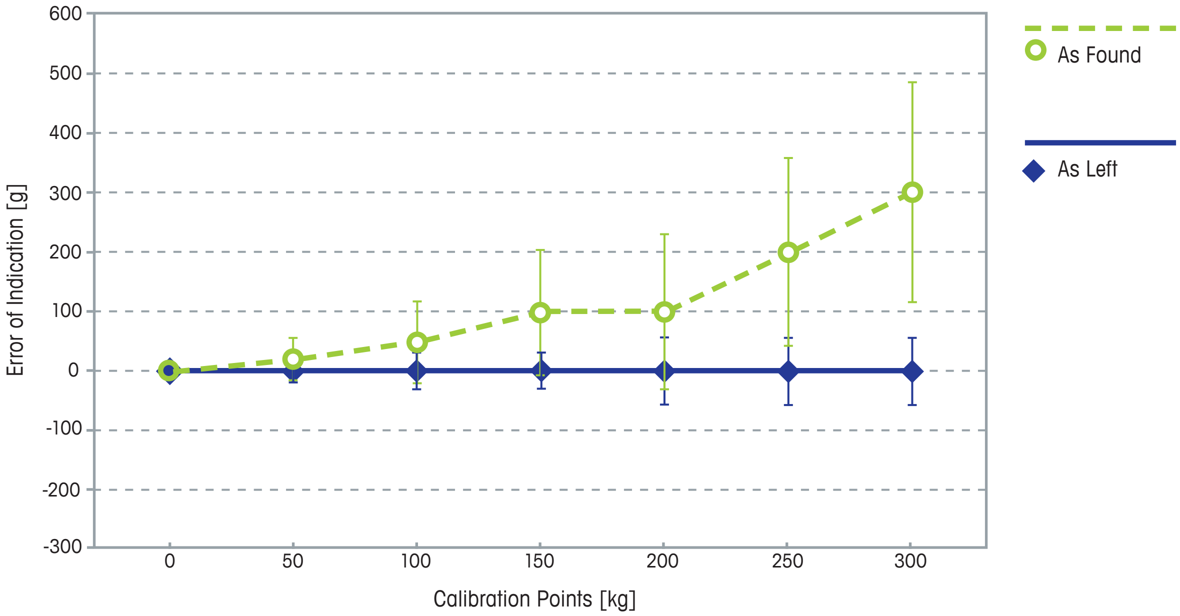

Figure 1: Graphical indication of the error of indication test points with their respective standard uncertainties from a calibration certificate (green circles for as found, before adjustment, and blue rhombi for as left, after adjustment of the scale).

The standard uncertainty $u(E)$ comprises of four individual contributions that are associated with the indication I, and four contributions which are associated with the reference mass m_{ref}. The uncertainty of the indication accounts for the rounding error of the no-load indication, the rounding error of the indication at load, the repeatability and the eccentricity of the weighing instrument. The standard uncertainty of the reference mass usually comprises of the tolerance or the uncertainty of the reference mass, air buoyancy, a possible drift of the reference mass since its last calibration and convection effects due to potential temperature differences between the reference mass and the instrument.

After having derived the standard uncertainty at calibration for the individual calibration points, these uncertainty values must be expanded by the coverage factor k which is chosen in a way such that the expanded uncertainty of measurement $U(E)$ has a coverage probability of 95.45 %, i.e. the expanded uncertainty shall ensure that the true value – which is not known – lies with a probability of at least 95.45 % within the interval 'measured value ± expanded measurement uncertainty'.

$$U(E) = ku(E)$$

Now, the calibration itself is formally completed. However, the data derived so far is of reduced value for the user as three sources of interpretation are missing:

- Behavior of the instrument in-between the selected error of indication test points
- Estimation of the measurement uncertainty in normal usage
- Assessment of the instrument against specific requirements such as weighing process tolerances

EURAMET cg-18 provides guidance on how estimate the measurement uncertainty for any quantity of material which is placed on the weighing instrument during normal usage. Introducing the parameters α_{gl} and β_{gl}, the so-called uncertainty of a weighing result $U(W)$ can be derived and approximated as a linear equation:

$$U(W) = \alpha_{gl} + \beta_{gl} \cdot R$$

with the offset α_{gl}, i.e. the uncertainty without load; the slope β_{gl}, i.e. the parameter describing the increase of the uncertainty when larger loads with an associated reading R are applied on the instrument.

3. Minimum Weight and Safe Weighing Range

It is general practice for users to define specific requirements for the performance of an instrument (User Requirement Specifications). Normally they define upper thresholds for measurement uncertainty values that are acceptable for a specific weighing application. Colloquially users refer to weighing process accuracy or weighing tolerance requirements. Typically these requirements are indicated as a relative value, e.g. adherence to a measurement uncertainty of 0.1%.

For a given tolerance requirement, Req, only weighings with a relative uncertainty $U(W)/R \leq Req$ fulfil the respective user requirement. The limit value, i.e. the smallest weighing result that fulfils the user requirement is called "minimum weight".

Due to specific environmental factors such as high levels of vibration, draughts, influences induced by the operator, etc., or due to specific influences of the weighing application such as electrostatically charged samples, magnetic stirrers, etc., a safety factor SF is usually applied. The safety factor is a number larger than one, by which the user requirement Req is divided. The objective is to ensure that the relative measurement uncertainty is smaller than or equal to the user requirement Req, divided by the safety factor.

This leads to the definition of the safe weighing range: It is the range of the instrument, where the user can weigh safely, i.e. he fulfils the weighing tolerance requirement and adheres to the defined safety factor, see figure 2.

Note that the minimum weight can vary between two subsequent calibrations due to potential performance changes of the instrument and varying environmental conditions. By means of application of an appropriate

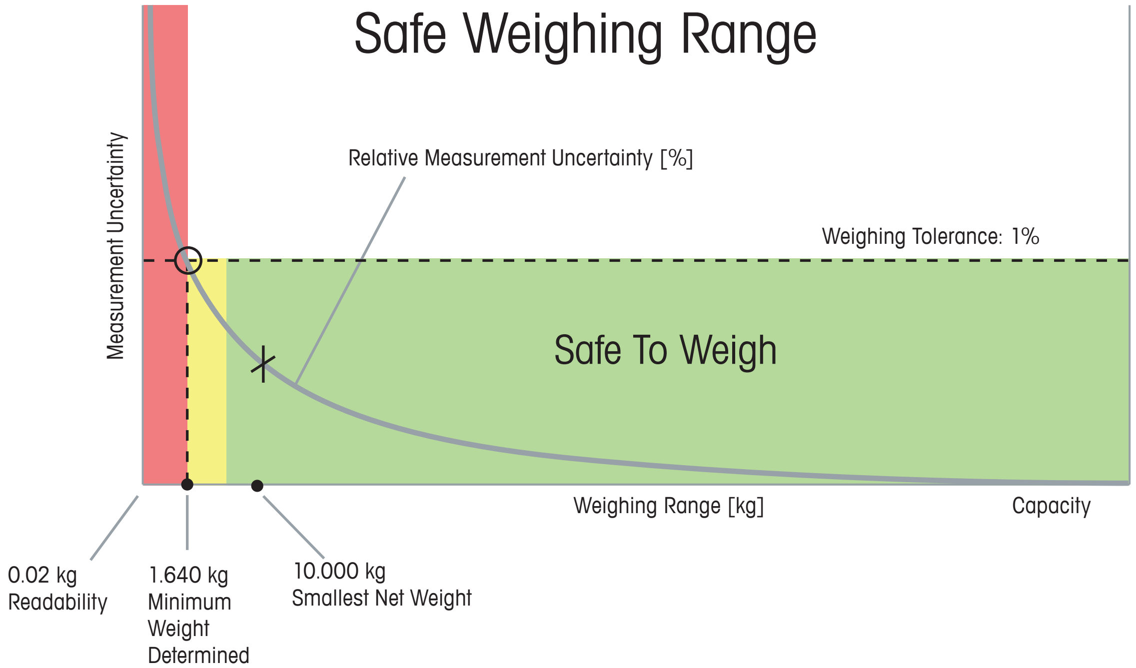

Figure 2: Safe weighing range for an industrial floor scale, derived from the calibration data and presented in an annex to a calibration certificate. The accuracy limit of the instrument, the so-called minimum weight, is the intersection point between relative measurement uncertainty and the required relative weighing tolerance. Weighing quantities in the red region results in non-compliance with the tolerance requirement, while weighing quantities in the green region ensures the tolerance requirement is fulfiled (safe weighing range). Weighing quantities in the yellow area fulfil the user requirements; however the safety factor is not adhered to.

safety factor it is ensured that the smallest net weight, i.e. the smallest net quantity that the user intends to weigh on the instrument, is always larger than the minimum weight whenever the instrument is used. Note that the minimum weight and the safe weighing range refer to the net (sample) weight which is weighed on the instrument, i.e. the tare vessel mass must not be considered to fulfil the user requirement Req. Therefore minimum weight is frequently called "minimum sample weight".

4. Conclusion

Calibration of measuring instruments is amongst the most important activities within any quality management system. Unfortunately, industry practices with respect to weighing instruments do not always appropriately reflect state-of-the-art concepts. The most evident shortcoming is the lack of a scientifically correct estimation of the measurement uncertainty, which is needed to assess whether the instrument under consideration fulfils predefined process tolerances.

The EURAMET cg-18 calibration guideline is the most widespread reference document that details the methodology of deriving the measurement uncertainty of non-automatic weighing instruments. It not only includes information on the uncertainty at calibration, but also on the uncertainty of a weighing result which describes the performance of the instrument during day-to-day work and frequently serves as a basis for assessing the instrument against predefined tolerances. A critical consequence of calibration is the concept of the minimum weight. By determining the minimum weight the user can assure compliance with his weighing requirements by weighing a sufficiently higher quantity of material than the minimum weight, expressed quantitatively by the safety factor. The minimum weight defines the lower boundary of the safe weighing range, and weighing quantities of material within the safe weighing range guarantees compliance with the required weighing process tolerance. In simple words, calibration and the subsequent interpretation of its data establishes minimum weight and safe weighing range, and ensures the user meets applicable quality requirements.

References

[1] JCGM 200:2012 (VIM), "International Vocabulary of Metrology – Basic and General Concepts and Associated Terms", Joint Committee for Guides in Metrology, 3rd edition with minor corrections, 2012.

[2] OIML R76-1, "Non-automatic weighing instruments, Part 1: Metrological and technical requirements – Tests", International Recommendation, Organisation Internationale de Métrologie Légale, 2006.

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[4] USP General Chapter 41, "Balances", US Pharmacopeia USP38 – NF33, United States Pharmacopeial Convention, 2015.

[5] EURAMET cg-18, "Guidelines on the Calibration of Non-Automatic Weighing Instruments", 3rd edition, European Association of National Metrology Institutes, March 2011.

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