

Consistency Test on Mass Calibration of Set of Weights in Class E₂ and Lower

Luis Omar Becerra, Ignacio Hernández, Jorge Nava, Félix Pezet

National Center of Metrology (CENAM)
Querétaro, Mexico

Abstract:

On weights calibration one by one there is the problem of checking the validity of the found values that could be deviated due to systematic errors e.g. a possible mass deviation of the weight standard used. The present work proposes a way to check the mass values resulting from the calibration that would support calibration decisions, especially applicable for the secondary calibration laboratories.

1. Introduction

On weights calibration there is frequently the problem of the truthfulness of the resulting values for the calibration.

Weights are delicate instruments to handle and it is common that the standard weights could drift the value of their mass, and there is not a way to note it before their next calibration.

It is proposed here a consistency test that offers the metrologist the possibility to perceive if the mass value of any of the standard weights is not consistent in the calibration of weight sets once the metrologist has an indication of non-consistency, he or she would have the possibility to take decisions as corrective actions.

2. Background

The International Recommendation OIML R111 [1,2] defines seven accuracy classes of weights from E₁ to M₃. The Recommendation OIML R111 defines the nominal values of mass standards as 1×10^n kg, 2×10^n kg or 5×10^n where “n” could be a negative number, positive number or zero, and the sequence of the sets could be formed from the next options (1;1;2;5) x 10ⁿ kg; (1;1;1;2;5) x 10ⁿ kg; (1;2;2;5) x 10ⁿ kg or (1;1;2;2;5) x 10ⁿ kg [1].

On weight calibrations of class E₂ and lower; weight standards of better class of accuracy are used, the mathematical model for the conventional mass value is the following [3],

$$m_x^c = m_p^c - (\rho_a - 1,2)(V_p - V_x) + \Delta m \quad (1)$$

Where,

m_x^c	Conventional mass of the unknown weight
m_p^c	Conventional mass of the standard weight
ρ_a	Air density
1,2	Conventional air density
V_p	Volume of the standard weight
V_x	Volume of the unknown weight
Δm	Mass difference between standard weight and the unknown weight read on the balance

The combined standard uncertainty of the conventional mass of the unknown weight is obtained from equation (2), [4]

$$u_{m_x^c} = \sqrt{u_{m_p^c}^2 + (V_p - V_x)^2 \cdot u_{\rho_a}^2 + (\rho_a - 1,2)^2 \cdot (u_{V_p}^2 + u_{V_x}^2) + u_{\Delta m}^2} \quad (2)$$

Where,

u_i Standard uncertainty of the variable “i”

The expanded uncertainty of the conventional mass is obtained by multiplying the combined standard uncertainty by a confidence factor, usually $k=2$ that amplifies the confidence intervals of the uncertainty to approximately 95%, the expanded uncertainty value must not be larger than 1/3 of the Maximum Tolerable Error (MTE) of the accuracy class of the calibrated weight, [1,2]

$$U_{m_x^c} = k u_{m_x^c} \leq \frac{1}{3} MTE \quad (3)$$

Where

$U_{m^c_x}$ The expanded uncertainty

k Coverage factor associate to the confidence level

3. Statistical control in mass calibration

A check standard is usually a weight of the same class and the nominal mass as the test weight to be calibrated and is included in the weighing design as an "unknown" weight. The control procedure works best with weighing designs (subdivision models) where the check standard can easily be incorporated into the design as an unknown weight.

The purpose of the check standard is to assure the goodness of calibrations. A history of values on the check standard is required for this purpose. The accepted value of the mass difference for the check standard (usually an average) is computed from the historical data and is based on at least 10 to 15 measurements and the value of the check standard for any new calibration is tested for agreement with the accepted value using a statistical control technique. The test is based on the **t-statistic** or **t-test**. [2].

For individual calibrations of weights one by one the use of the check standard may not be recommended.

By other hand the precision of the balance can also be monitored using a statistical control technique. The residual standard deviation from weighing design or a standard deviation of repeated measurements on a single weight is the basis for the test. The test relies on a past history of standard deviations on the same balance. If there are m standard deviations of the balance from historical data from which a pooled standard deviation is obtained, the statistical test consists in comparing the value of F obtained from the new "within-group standard deviation" and the pooled standard deviation, versus the corresponding F critical value (according to the degrees of freedom), **F-test** [2].

The use of **F-test** indicates if the dispersion of the differences between the weight standard and the weight unknown obtained from the balance, is within the permissible limits (based in historical data), but this test is not sensible to

other possible sources of error as, for example, a possible drift of the standard.

4. Consistency Test

The consistency test consists in a calibration of a group of weights of a particular decade, (all together as an unknown weight), using the corresponding weight standard equivalent. The sum of the individual values of the weights should be equal to the value found on the calibration of the group of weights between the uncertainty values.

For example in a set of weights of nominal values from 100 g to 500 g, the weights 100 g, 200 g, 200 g(*), and 500 g are calibrated one by one and finally all weights together as a group of 1 kg as nominal value is calibrated versus a 1 kg weight standard, see Table 1.

Standard Weight		Unknown Weight
100 g	Vs	100 g
200 g	Vs	200 g
200 g	Vs	200 g(*)
500 g	Vs	500 g
1 kg	Vs	100 g + 200 g + 200 g(*) + 500 g

Table 1. Scheme of comparisons for 100 g to 500 g

The difference between the mass value of the group of weights and the sum of the individual mass values obtained should satisfy the following criterion that is used to verify equivalence of results in measurement comparisons among laboratories in [5] and [6],

$$E_n = \frac{|m^c \sum m^c_i - \sum m^c_i|}{\sqrt{\left(U_{m^c \sum m^c_i}\right)^2 + \left(U_{\sum m^c_i}\right)^2}} \quad (4)$$

$E_n \leq 1$ Found mass values of the calibration are consistent, the calibration would be considered correct.

$E_n > 1$ Found mass values of the calibration are not consistent between then, it is necessary take corrective actions.

Where,

E_n Normalized error value

$m^c \sum m^c_i$ Conventional Mass of the group of the weights (obtained from the equation 1, taking as unknown weight the set of weights)

$\sum m^c_i$ Sum of the conventional masses found on its individual calibration

$U_{m^c \sum m}$ Expanded uncertainty obtained on the group calibration accord with the equation 3

$U_{\sum m^c_i}$ Sum of the mass expanded uncertainties from the individual calibration of the weights, equation 5,

$$U_{\sum m^c_i} = \sum U_{m^c_i} = k \sum u_{m^c_i} \quad (5)$$

where the correlation coefficient between the values of the conventional mass of the weights is taken as 1 [7].

5. E_n Criterion

A meaningful comparison of two results of measurement of the same quantity requires the statement of the uncertainties of the measurements since they characterize the reliability of the measurement. The "normalized error" (E_n) takes into account of this. Here the sum of masses provides a value to compare it with the mass of the group of weights. The expanded uncertainties associated with the results of measurement are denoted with $U_{\sum m^c_i}$ and $U_{m^c \sum m}$ result from the multiplication of the standard uncertainties by the same fixed coverage factor $k > 1$ that usually is taken to be 2. For $k=2$, often the compatibility criterion $E_n \leq 1$ is proposed [5]. This is equivalent to the procedure for comparing materials or products with respect to average performance developed in [8].

It is clear that a significant difference between $m^c \sum m^c_i$ and $\sum m^c_i$ indicates the inappropriate treatment of the systematic effects by at least one of the measurements. Thus, in this case at least one of the quoted results of measurements is not reasonable estimate of the value of the measurand but may be viewed as the

best estimate of the value of a different quantity. Therefore, if $m^c \sum m^c_i$ and $\sum m^c_i$ are taken as best estimates of the unknown values of the Conventional Mass of the group of the weights ($M^c \sum m^c_i$) and the Sum of the Conventional Masses ($\sum M^c_i$), respectively, they may be regarded to meaningfully represent the same quantity if the available data support the hypothesis that the difference $D = M^c \sum m^c_i - \sum M^c_i$ is zero.

The best estimate of D is $d = m^c \sum m^c_i - \sum m^c_i$ and the standard uncertainty associated with d is

$$u_d = k \left[\left(u_{m^c \sum m} \right)^2 + \left(u_{\sum m^c_i} \right)^2 \right]^{1/2} \text{ in case of}$$

the assumed independent measurements. The values that can reasonably be attributed to D lie within the limits $d \pm u_d$. If the value zero is within the limits the acceptance of $D = 0$ is not in conflict with the available information about $M^c \sum m^c_i$ and $\sum M^c_i$, and, thus, D . In this case the values of $m^c \sum m^c_i$ and $\sum m^c_i$ can reasonably be viewed as two differently established estimates of the value of the same quantity and, hence, as compatible.

6. Numeric Example

On a set calibration are obtained next conventional mass values on weights from 100 g to 500 g

Nominal Value	Correction* mg	Uncertainty (k=2) mg
100 g	+ 0,153	±0,027
200 g	-0,006	±0,032
200 g	-0,003	±0,032
500 g	-0,016	±0,044
$\sum m^c_i$	0,128	±0,135
$m^c \sum m^c_i$ (1 kg)	-0,021	±0,174

Table 2. Conventional Mass Values found in calibration in a set weight form 100 g to 500 g

*Correction is equal to the conventional mass less to the nominal value of the weight

The normalized error is obtained from equation 4,

$$E_n = \frac{|m^c \sum m^{c_i} - \sum m^c_i|}{\sqrt{\left(U_{m^c \sum m^c_i}\right)^2 + \left(U_{\sum m^c_i}\right)^2}} = 0,68$$

The value of E_n is lower than 1, it means that the values of the calibration are consistent between them (Fig. 1). The value of E_n indicates that none of the standards used on the calibration have drifted (the only possibility is that all of them have drifted together), the calculation of the conventional mass and the uncertainty looks correct.

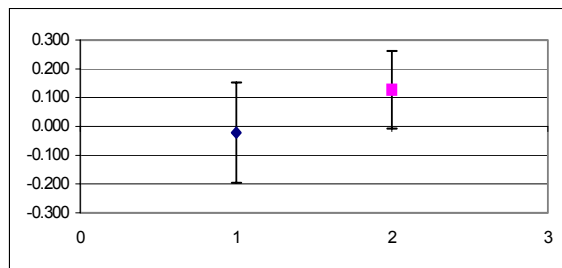


Figure 1. Comparison between conventional mass corrections of $m^c \sum m^{c_i}$ and $\sum m^c_i$ and its uncertainties respectively

Now let see other example where the values obtained in the calibration are on table 3.

Nominal Value	Correction Mg	Uncertainty (k=2) mg
100 g	+ 0,153	±0,027
200 g	+ 0,080	±0,032
200 g	-0,003	±0,032
500 g	-0,021	±0,044
$\sum m^c_i$	+ 0,209	±0,135
$m^c \sum m^{c_i}$ (1 kg)	- 0,021	±0,174

Table 3. Conventional Mass Values found in calibration in a set weight form 100 g to 500 g

The normalized error has next value,

$$E_n = \frac{|m^c \sum m^{c_i} - \sum m^c_i|}{\sqrt{\left(U_{m^c \sum m^c_i}\right)^2 + \left(U_{\sum m^c_i}\right)^2}} = 1,04$$

E_n on this example is larger than 1, and it means that values found on the weights calibration are

not consistent between them, although the uncertainty bars touch between them (Fig 2).

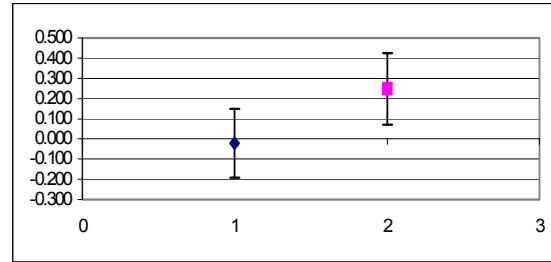


Figure 2. Comparison between conventional mass correction of $m^c \sum m^{c_i}$ and $\sum m^c_i$ and its uncertainties respectively

This situation gives us an alert message about the results of the calibration. This offers us a chance to check the calibration process (data transferring, calculus, calibration of the standards, sensors, etc), before to submit a calibration certificate.

If the mass laboratory has a history of E_n values for different decades a statistical analysis could be done from these values and establishing the intervals of calibration based on them.

7. Discussion

The consistency test could be applied to any accuracy class of weight, but it is recommended for class E_2 and lowers because for mass calibration of weights class E_1 are used models of subdivision where check standards are introduced in the models for the statistical control. For accuracy classes lowers than E_1 the consistency test could offer confidence to the mass laboratory on their certificates and for the customs for the service received.

It is necessary to say that this test is not sensitive to an equal drift in all standards involved in a calibration, because the results obtained would be consistent between them, but may deviates from the real value; this possibility is avoided with the calibration program of the standard.

On mass calibration of weight classes E_1 and E_2 , normally the measurement of the volume of the weights is required. The volume is usually measured by hydrostatic weighing and the consistency test could be adequate to volume measurements where the sum of volumes could be compared versus the volume of the group of weights (normally a decade).

8. Conclusions

The use of the normalized error consists in comparing the difference between the conventional mass of the group of weights and the sum of the conventional masses of individual weights in the range of the combined uncertainty of both calculations. If the mass difference between both calibrations is small than the combined uncertainty of both values, then the mass calibrations is acceptable, but if the mass difference is large than the combined uncertainties then there is a problem in the calibrations and the values of the uncertainties obtained do not cover it.

The use the consistency test could complementary to the F-Test, because the goal of the F-Test is to check if the standard deviation of the balance was acceptable.

It is important to say that the uncertainty has a limit (1/3 MTE), and from this point of view there is not possibility to enlarge the uncertainty in order to cover this difference between the two values of mass.

The consistency test could be a useful tool for the metrological assurance of the mass laboratories, because with the E_n values the laboratory could define calibration intervals and support all values declared on certificates or reports of calibration. The confidence of the customs could increase about the service required knowing that E_n values obtained on the calibration of their weights were less than a chosen value.

On evaluations of technical capability of Mass laboratories could be useful for the auditors checking the values of E_n obtained on routine calibration services.

9. References

- [1] OIML, R111 International Recommendation N° 111 -Weights of classes E_1 , E_2 , F_1 , F_2 , M_1 , M_2 , M_3 , 1994
- [2] OIML, Draft International Recommendation N° 111 - Weights of classes E_1 , E_2 , F_1 , F_2 , M_1 , M_2 , M_3 , (including weights for testing of high capacity weighing machines) Part :1 Metrological and Technical Requirements- February 2000

- [3] OIML, R33 International Recommendation N° 33 Conventional Value of the result of weighing in air", 1979
- [4] BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML, "Guide to the expression of uncertainty in measurement" Corrected and reprinted, 1995
- [5] Wolfgang Wöger -Remarks on the E_n - Criterion Used in Measurement Comparison, PTB-Mitteilungen 109 1/99, Internationale Zusammenarbeit
- [6] European cooperation for Accreditation of Laboratories -EAL Interlaboratory Comparisons- (March 1996)
- [7] Bich W., 1990, "Variances, Covariances and Restraints in mass metrology", Metrologia 27, 111-116 (1990)
- [8] Natrella G. Mary, -Experimental Statistics; Handbook 91 -1963 -National Institute of Standards and Technology

Contact Person for Paper:

M. in Sc. Luis Omar Becerra
Mass and Density Division
Address: km. 4,5 Carretera a los Cués Mpio. El Marqués; Querétaro, México C.P. 76900
Fax: (+52) 4 2 15 39 04
Phone: (+52) 4 2 11 05 00 to 04 ext. 3602
e-mail: lbecerra@cenam.mx