Integral comparison approach for standards weight collections checking

A Beatrici
Instituto Nacional de Metrologia, Qualidade e Tecnologia - Inmetro. Rio de Janeiro, BR.
E-mail: abeatrici@inmetro.gov.br

Abstract. The standard collections checking are a periodic laboratory procedure that are needed to keep (and to extend) the validation of the mass standards calibration certificate data to provide metrological traceability in their own measurement results. In this study will be shown a full comparison procedure of a mass standard set, by an equations system with n interconnected nominal values. Indeed this procedure intents to improve the quality assurance and to reduce costs to the laboratories that needs to keep calibrated mass standards collections. The main approach advantage is do not need another reference standards set.

Keywords: mass metrology, weigh checking, mass calibration.

1. Introduction
Checking, or verifying, standard weight collections is a periodic activity in laboratories requiring the use of reference mass standards for balances calibrations [1] [2] [3] or standards weights collections calibrations. These checking are usually done on just some nominal values to attest the complete standards set stability. A typical kilogram submultiples standards weights collection have nominal values from 1 mg up to 1 kg, including the repeated nominal values, which are usually multiples of the unit, or can be multiples of 2, having a total of 25 standards per collection, while the kilogram multiples, due to their cost and size, are purchased individually and have nominal values of 2, 5, 10, 20 and 50 kg [1]. Both checking, kilogram multiples and submultiples, are done through directly comparing each nominal value with another standard from a higher accuracy class mass standards collection or at least more stable standard collection with better material physical characteristics, better finishing, lower measurement uncertainty and even lower exposure to the factors that may change its mass. In this procedure, there is the limitation of checking only some of the 25 standards, to the kilogram submultiples case, which makes it possible for one, or more standards in this collection, to have drifted its mass value and not be checked, but be assumed as stable ones. Another limitation is the need to have another better accuracy mass standard collection, which is not always available in the laboratory. Both limitations can be bypassed using an integral comparison procedure. This approach consists of the comparing each collection nominal value with the sum of all remaining minor nominal values of the same collection. Indeed in this approach we have a system of \( n \) equations interconnecting all of standard weights in the collection, preventing any mass standard from being considered stable without being properly checked. This system of \( n \) equations is constructed by an upper diagonal
matrix plus the acceptances criteria for the mass standard collection stability, so it is easily possible to find, through a row vs column analysis, which standard weights are outside of the verification criterion (check) or if all standards reach the established criteria for checking. The approach limitations are to need an automatic mass comparator, since its lack makes integral comparison extremely complicated and exposing the standard weights to an excessive handling risk.

The first equation, the full kilogram, requires a 200 g support disc or any other nominal value (existent in the mass standard collection), since it can be able to support the standards weights combination and that have been calibrated because its value will enter in the final equation. On the other hand, the advantage of using full comparison is the smaller number of comparisons and verifications to ensure the whole mass standard collection stability in only one procedure.

2. Integral approach equations

If we are in a hypothetical situation, where the standard collection to be checked has the table 1 calibration data, where \( NV \) is the standard weight nominal value, \( m_c \) is its conventional mass and \( U \) is the measurement-expanded uncertainty. To facilitate the equations understanding there is a column where the name of each variable is defined.

**Table 1.** Calibration certificate data and variable identification.

| \( NV \) (g) | Variable label | \( m_c \) (g) | \( U \) (g) |
|------------|---------------|--------------|-----------|
| 0.001      | \( n1 \)      | 0.001020     | 0.000002  |
| 0.002      | \( n2 \)      | 0.001930     | 0.000002  |
| 0.005      | \( n5 \)      | 0.005090     | 0.000002  |
| 0.010      | \( n10 \)     | 0.009910     | 0.000003  |
| 0.020      | \( n20 \)     | 0.019930     | 0.000003  |
| 0.050      | \( n50 \)     | 0.050420     | 0.000004  |
| 0.100      | \( n100 \)    | 0.101820     | 0.000005  |
| 0.200      | \( n200 \)    | 0.200070     | 0.000060  |
| 0.500      | \( n500 \)    | 0.499430     | 0.000080  |
| 1.000      | \( m1 \)      | 1.002450     | 0.000010  |
| 2.000      | \( m2 \)      | 2.000090     | 0.000012  |
| 5.000      | \( m5 \)      | 5.000790     | 0.000015  |
| 10.000     | \( m10 \)     | 9.999320     | 0.000020  |
| 10.000     | \( m10P \)    | 10.003190    | 0.000020  |
| 20.000     | \( m20 \)     | 20.003130    | 0.000025  |
| 50.000     | \( m50 \)     | 49.021620    | 0.000030  |
| 100.000    | \( m100 \)    | 99.999560    | 0.000050  |
| 100.000    | \( m100P \)   | 100.021990   | 0.000500  |
| 200.000    | \( m200 \)    | 200.071230   | 0.001000  |
| 500.000    | \( m500 \)    | 500.159870   | 0.002500  |
| 1000.000   | \( m1000 \)   | 1000.348000  | 0.005000  |
| 200.000    | \( DC200 \)   | 200.013500   | 0.001000  |
| 0.001      | \( milli \)   | 0.001009     | 0.000002  |

Using an automatic mass comparator and support disks, with at least one of these calibrated, the full comparison can be applied.

There is still another condition, the manufacturing material from 1 g to 1 kg standards need to be the
same, usually austenitic stainless steel, but in this approach really the condition of the same manufacturing material is enough to be possible to check the mass standards collections of any material and accuracy class. This condition is important because the integral comparison perform with the same material (density) suppresses the buoyancy effects corrections, or at least reduce it dramatically.

The first equation compares 1 kg weight with the 1 kg obtained by the sum of other mass standards of the same standard collection and one support disk, for example the DC200 (see table 1). The kilogram equation is the only one that needs a calibrated support disc, the others need the support discs without calibration or defined nominal value because the two-way comparison can be done, direct and inverted, thus eliminating the influence of the support disc mass on the measured mass difference. Actually the support disc just need to have the same density e approximately the same mass, but if two calibrated support discs are available it will be simpler to write the following equations. To simplify, it is convenient to group all the milligram standards (less than 1 g) so that the equation is in a more compact form, so we have this in the equation (1),

\[ N_{mg} = n_1 + n_1P + n_2 + n_5 + n_{10} + n_{10P} + n_{20} + n_{50} + n_{100} + n_{100P} + m_{200} + m_{500} \]  

(1)

The nominal value sum in equation (1) left term is exactly 0.999 g. Thus, we can write the two first kilogram equations (2) and (3),

\[ m_{1000} = N_{mg} + m_1 + m_1P + m_2 + m_5 + m_{10} + m_{10P} + m_{20} + m_{50} + m_{100} + m_{100P} + m_{200} + m_{500} \]  

(2)

\[ m_{1000} = N_{mg} + m_1 + m_1P + m_2 + m_5 + m_{10} + m_{10P} + m_{20} + m_{50} + m_{200} + m_{500} + DF_{1000} \]  

(3)

Where \( DF_{1000} \) and \( DF_{1000P} \) are the mass differences measured in the respective integral comparison. In equations (2) and (3) there is a difference of 1 mg in the integral comparison, this can be bypassed by placing an extra standard of 1 mg on the right term in equation (1), if available, or just subtracting the 1 mg nominal value on the left term in all of integrals comparisons equations except for the last equations that comparing 2 mg and 1 mg. The following equations are in descending order of nominal values corresponding to 500 g down to 10 g, equations (4) to equation (13).

\[ m_{500} = N_{mg} + m_1 + m_1P + m_2 + m_5 + m_{10} + m_{10P} + m_{20} + m_{50} + m_{100} + m_{100P} + m_{200} + DF_{500} \]  

(4)

This equation, and the following ones, in the case of not availability of two calibrated support discs of the same nominal value, may be modified by including tare disc on each side of its equations. Here we choose to use the direct and inverted comparison to eliminate the influence of the support discs.

\[ m_{200} = N_{mg} + m_1 + m_1P + m_2 + m_5 + m_{10} + m_{10P} + m_{20} + m_{50} + m_{100} + DF_{200} \]  

(5)

\[ m_{200} = N_{mg} + m_1 + m_1P + m_2 + m_5 + m_{10} + m_{10P} + m_{20} + m_{50} + m_{100P} + DF_{200P} \]  

(6)

\[ m_{100} = N_{mg} + m_1 + m_1P + m_2 + m_5 + m_{10} + m_{10P} + m_{20} + m_{50} + DF_{100} \]  

(7)

\[ m_{100P} = N_{mg} + m_1 + m_1P + m_2 + m_5 + m_{10} + m_{10P} + m_{20} + m_{50} + DF_{100P} \]  

(8)

\[ m_{50} = N_{mg} + m_1 + m_1P + m_2 + m_5 + m_{10} + m_{10P} + m_{20} + DF_{50} \]  

(9)

\[ m_{20} = N_{mg} + m_1 + m_1P + m_2 + m_5 + m_{10} + DF_{20} \]  

(10)
\[ m_{20} = Nmg + m1 + m1P + m2 + m5 + m10P + DF20P \]  
\[ m_{10} = Nmg + m1 + m1P + m2 + m5 + DF10 \]  
\[ m_{10P} = Nmg + m1 + m1P + m2 + m5 + DF10P \]

Where all variables beginning by \( DF \) at the end of each equation, represent the respective physically measured mass differences in the respective integral comparison equation.

The next equations (14) through (17) must be careful analyzed with respect to the resolution of the used equipment in relation to the accuracy class requirements. For example, if we use a collection of accuracy class \( E_1 \) on a one microgram resolution mass comparator, the next equations may not reach the resolution criteria required for the checking. Mostly \( E_2 \) or lower accuracy class can usually performed up to 100 mg, but to the lower nominal values, this situation must studied to be possible to perform the comparison, but in this case, a better resolution mass comparator will be needed [4].

\[ m5 = Nmg + m1 + m1P + m2 + DF5 \]  
\[ m2 = Nmg + m1 + DF2 \]  
\[ m2 = Nmg + m1P + DF2P \]  
\[ m1 = Nmg + DF1 \]

The milligram weights equations \((Nmg)\) follows the same construction pattern, with the measured differences from the integral comparison being represented by lowercase \( df \) to avoid variables misunderstanding. Since the assembly of equations are similar to the previous ones, there is no needs to specify them at this time. In the same way the kilogram multiples equations do not need to be shown either the others arrangements of integral equations to the others kinds of mass standard collection commercial distributions.

The first time application of this approach is an extensive procedure because it is necessary to perform all these integral equations weighing to identify the values of the integral differences \( DF \) and \( df \) of each nominal value. In the following applications only the equations \( m1000, m500, m200, m50, m20, m2 \) and \( m1 \), so with few equations we can check the whole standards collection periodically, because there are previous established integral differences data, thus improving the quality control in the laboratory measurement results. In fact, only the first integral equation, the kilogram equation, already provides a good stability indication, or instability, of the collection in later checks.

After the spreadsheet data filling, the conventional mass data, their respective measurement expanded uncertainties and the measured differences of the balance indications results for each equation, is too simple to perform the comparison, we need just to compare the mass differences calculated (from the calibration certificate data) with the measured mass differences (balance indication differences in the physical comparison process). The acceptance criterion adopted in this case is that these values differences must be less than \( 1/3 \) of the maximum mass error value of the used standards accuracy class. This uncertainty value is that of each standard used for assembling the equation, for example in the kilogram equation is used the kilogram uncertainty, in the 500 g equation the 500 g measurement uncertainty, and so on. This criterion is easily changed according to the user requirements.

3. Standards positions in weighing procedure
To mitigate the center mass distribution effects due to the height difference between both sides of the
integral mass comparisons, it is suggested that a mass standard distribution such that minimize this difference. In the case of $E_2$ accuracy class standards (or lower), this is not critical, but in the case of accuracy class $E_1$ care must be taken. Table 2 shows the standards size to allow the mass center calculation.

### Table 2. Typical height values for stainless steel standards.

| NV (g) | Height (mm) |
|--------|-------------|
| 1000   | 81          |
| 500    | 63          |
| 200    | 47          |
| 100    | 39          |
| 50     | 30          |
| 20     | 22          |
| 10     | 18          |
| 5      | 14          |
| 2      | 10          |
| 1      | 6           |

With these data, we can easily simulate the distribution that minimizes the mass center difference. All distributions of minimum mass center differences can be simulated to avoid the need of this correction. This procedure needs to be performed just once time per mass standard collection.

For one kilogram mass standard we have around 3 micrograms per centimeter of mass center correction. With equal or approximately equal center mass distribution this correction can be lowered than the balance resolution. In any case of the integral comparison we can place the standards in a condition to minimize the mass center correction.

![Figure 1.](image)

To the weights smaller than 1 g the standards can be distributed on the available space. The buoyancy effect can be neglected, since is assumed that the density are homogeneous in the whole mass standard collection. It is a very reasonable supposition but can be assured by the direct volume measurement or by testing the measurements results to different environmental conditions checking if there is some distinct buoyancy effect.
4. Results

The results calculations will be shown based on the calibration results presented in table 1. The mass drift values of the standards are simulated, so that it is possible to verify how to identify the standard weight that shows the drift condition. In the implemented example all integrals equations for a 25 standard collection for 1 mg up to 1 kg were performed. The present version use an extra 1 mg standard from another collection, but as described earlier this can be easily bypassed if an extra standard is not available. In the absence of a calibrated support disc, the first equation for full 1 kg comparison cannot be performed.

After concluded the integral comparisons and determined its indications differences $DF$ and $df$ for each equation, the differences between the certificate and integral comparison data are performed. The criterion for each equation is a variation of up to 1/3 of the maximum error of each nominal value for a given accuracy class, in this case E2 accuracy class standards. To exemplify the scope of this approach we simulate two cases of standards that are out of calibration.

In the first case changing the standard $m10P$ mass by a - 20 micrograms drift in its mass value, as soon as these data is fulfilled in the spreadsheet, the FALSE indication appears in two equations in the nominal value column (figure 2 and 3), $m10P$ and $m20$. Crossing the $mp10$ line with the $mp10$ column and the same for the $m20$, it is easy to see that only the $m10P$ standard appears in both equations with the FALSE affirmation. This can be verified by a new calibration of these mass standards.

![Table showing integral comparison results](image)

**Figure 2.** Integral comparison simulated test to 20 micrograms drift.
Figure 3. Integral comparison simulated test to 60 micrograms drift.

In the second case (Figure 3), for the same standard, it was drifted by -60 micrograms. Using the same row vs. column intersection analysis it is easy to identify the standard that appears in all equations encoded as FALSE. To the most complicated case, with several drifted mass standards, may be necessary to perform two or more line vs. column analysis, but this is not a common occurrence in research laboratories.

5. Conclusion
This approach is a good alternative procedure to ensure the fast and easy identification of the mass standard drift. The advantages can be summarised by:

i) To reduce or to eliminate the buoyancy effect by using of the same density mass standards.

ii) To reduce or to eliminate the mass center corrections by the mass standards distribution.

iii) To identify easily the drifted mass standard.

iv) To check all mass standards in the same procedure.

v) To make the checking with no requirement of another high accuracy class mass standards collection.

vi) To change the acceptances criteria easily.

vii) To create or adopt individual’s acceptances criteria to test each integral equation is very simple.

viii) To reduce the laboratory operational costs.
The disadvantages are the difficulties to place the 1 kg integral comparison mass standards, this extensive procedure, in the first integral comparison perform, require an automatic mass comparator and the at least one calibrated disc support available.

Actually we can check the mass standard collection stability only comparing the main check equations because its mass differences stability ensures the stability condition to the whole mass standard set.

Finally, this checking approach can be also applied as a way to perform subdivision or multilication method. To this will be necessary to develop and to apply a variational adjustment method to provide the better results of the mass unit dissemination, the kilogram.

References
[1] OIML Recommendation R 111 (Parts-1 and 2) 2004. Weights of classes E₁, E₂, F₁, F₂, M₁, M₁-2, M₂, M₂-3 and M₃. International Organization of Legal Metrology.
[2] OIML D28 2004. Conventional value of the result of weighing in air. International Organization of Legal Metrology.
[3] EURAMET/cg-18 (version 4.0) 2015. Guidelines on the Calibration of Non-Automatic Weighing Instruments. Euramet.
[4] Technical Report 2015. Validation of the calibrations procedure using adjusted mass disc supports. Dimci R – 1898/2015 (Internal document). Inmetro, Brazil.