Ensuring the validity of results by intermediate checks in the field of mass measurements

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Abstract. In 2017, the third edition of ISO/IEC 17025 standard was completed. This version will cancel and replace the second edition from 2005, which has been technically revised. Even if, in the second version, maintaining confidence in the calibration status of standards through intermediate checks was not clearly explained in terms of a procedure (referring to this term only in subchapters 5.5.10, 5.6.3.3 and indirectly in 5.9.1), nor in the new version of the standard this subject is not detailed. It is up to each laboratory to carry out these checks according to defined procedures and schedules as well establishing the acceptance criterion. So, the questions arising about performing intermediate checks are: when, where, how? The paper presents different approaches regarding intermediate checks performed in the field of mass measurements: in the calibration/verification of weights, of mass comparators and NAWI (non automatic weighing instruments). In the case of NAWI, considering the laboratory’s scope of accreditation, the evaluation method is presented in different ways.

1. Introduction
Intermediate checks are performed to maintain confidence in the calibration status of measuring and test equipment between calibrations [1]. When the results of intermediate checks indicate that the measuring and test equipment is no longer performing in accordance with the specified requirements, the laboratory shall either shorten the intervals between calibrations or taken other appropriate corrective actions. In this context, the key questions arising about intermediate checks are: when, where and how should be performed? The answers could be:

- When: intermediate checks should be performed periodically, between calibrations. The periodicity should be defined in a reasonable way.
- Where: in own laboratory, using proper reference standards.
- How: regardless of the method used in performing intermediate checks, in the procedure it is necessary to establish an acceptance criterion. If the acceptance criterion is exceeded during intermediate check, the laboratory should take proper corrective actions.

The paper presents different approaches regarding intermediate checks performed in the field of mass measurements: in the calibration/verification of weights, of mass comparators and NAWI. In the case of NAWI, considering the laboratory’s scope of accreditation, the evaluation method is presented in different ways.

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2. Intermediate checks used in the calibration/verification of weights

Generally, intermediate checks in the field of calibration/verification of weights are applied in the dissemination of mass unit. For this purpose, for ensuring the validity of results by intermediate checks, it can be used either a simply calibration of the weight by Borda method, (the acceptance criterion being the normalized error, \(E_n\)), either using a combination, which consists in introducing the respective weight in the measurement scheme as a check standard (the acceptance criterion being either the normalized error, \(E_n\), either a statistical control process). This second method is used in the calibration of \(E_1\) class, where additional weights should be introduced in the matrix design. In this way, the mass value obtained for check standards can be considered as intermediate checks for the respective weights. If the values obtained from the individual calibrations are to be compared with those from calibration certificate, a suitable acceptance criterion can be calculated, using so-called \(E_n\) value [2].

\[
|E_n| = \frac{\delta_{IC} - \delta_{certif}}{\sqrt{U_{IC}^2 + U_{certif}^2}} \leq 1
\]  

\(\delta_{IC}\) represents the mass error of the weight obtained during intermediate check; 
\(\delta_{certif}\) the mass error of the weight from the calibration certificate; 
\(U_{IC}\) the expanded uncertainty of the weight obtained during intermediate check; 
\(U_{certif}\) the expanded uncertainty of the weight from the calibration certificate.

Using this formula, the results obtained during intermediate check is considered acceptable if the value of \(E_n\), is between -1 and +1. When more results for the same weight are available (generally for class \(E_1\) weights, but not only), it can be used a statistical control. For this purpose, a history of mass values for the weight used as check standard is required. In Table 1 are presented the results obtained from 10 intermediate checks of the same weight of 100g, class \(E_1\) (considered as check standard), during a period of one year and also the value obtained from the last calibration.

Table 1. The results of intermediate checks (IC) for a weight of 100g.

| No of IC | Mass error value for weight of 100g | No of IC | Mass error value for weight of 100g | “t” | Critical value of Student’s \(t\)-distribution |
|---------|-------------------------------------|---------|-------------------------------------|------|-----------------------------------------------|
| 1       | 0.074 mg                            | 6       | 0.028 mg                            |      |                                               |
| 2       | 0.067 mg                            | 7       | 0.070 mg                            |      |                                               |
| 3       | 0.090 mg                            | 8       | 0.065 mg                            | 0.156| 2.262                                         |
| 4       | 0.061 mg                            | 9       | 0.052 mg                            |      |                                               |
| 5       | 0.057 mg                            | 10      | 0.061 mg                            |      |                                               |

\(\bar{m}_{diff} = 0.0625\) mg; \(s = 0.0160\) mg; Last IC \((m_{diff}) = 0.065\) mg

The value obtained at the last intermediate check for the respective weight (which was incorporated into the design scheme as check standard), \(m_{diff}\), is tested for agreement with the accepted value. For this purpose, the \(t\)-statistic, in accordance to [3] is calculated:

\[
t = \frac{|m_{diff} - \bar{m}_{diff}|}{s}
\]  

where: \(s\) is the standard deviation of \(n\) historical values of the mass differences, having \(\nu-1\) degrees of freedom:

\[
s = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (m_{diff} - \bar{m}_{diff})^2}
\]  

As it can be seen from the Table 1, the evaluation process is judged to be in control because: \(t \leq\) critical value of Student’s \(t\)-distribution, with \(\nu = 9\) degrees of freedom.

3. Intermediate checks in the field of mass comparators

There are two ways to use a weighing instrument: as a direct weighing instrument (a common balance) and as a mass comparator, in the dissemination of mass unit from national mass standard (with values derived from the International Prototype of the Kilogram) to the standards in routine use. For this purpose, the following
parameters of the mass comparator are determined and listed in the calibration certificate: sensitivity and/or the mass value of the smallest scale interval, measurement repeatability results and, if necessary, the effect of the loads eccentrically placed (eccentricity). The mass comparator gives the difference of mass values between two weights (mass standard and test weight) using a differential weighing method (Borda - substitution method or Gauss- transposition method). In the intermediate checks, the most important characteristic that is being evaluated is standard deviation \( s \) (determined according to measurement cycles used, ABBA or ABA).

\[
s = \sqrt{\frac{\sum_{i=1}^{n} (\Delta_{\text{med}} - \Delta_i)^2}{n-1}} \tag{4}
\]

Where: \( \Delta_i \) represents the mass difference obtained from one measurement cycle and \( \Delta_{\text{med}} \) is the average of \( n \) measurement cycles. It is not mandatory to perform an individual determination of the repeatability for a mass comparator, but either the residual standard deviation of a weighing design (design matrix) or the standard deviation obtained from repeated measurements performed in the calibration of a weight, can be used. The test is based on a history of standard deviations performed on the same balance, at the same load [3].

\[
s_p = \sqrt{\frac{1}{m} \sum_{i=1}^{m} s_i^2} \tag{5}
\]

With: \( m \) number of standard deviations, \( s_1\ldots s_m \) and \( s_p \) is the pooled standard deviation having \( m \cdot \nu \) degrees of freedom. Each individual standard deviation has \( \nu \) degrees of freedom. The test statistic assumes that:

\[
F = \frac{s_{\text{new}}^2}{s_p^2} \leq \text{critical value from the } F\text{-distribution.} \tag{6}
\]

If this relation is valid, then the precision of the balance is judged to be in control. Each new standard deviation obtained either using a weighing design, or from repeated measurements performed in the calibration of a weight are tested against the pooled standard deviation.

4. Intermediate checks in the field of NAWI

The intermediate checks for these weighing instruments can be performed in different ways, considering the laboratory’s scope of accreditation.

4.1. Laboratories accredited for the calibration of NAWI

Considering that a calibration of NAWI is performed generally every 2 years (sometimes yearly), the intermediate check should be performed at the midpoint of the calibration interval. A “mini” calibration (for example in three points), according to [4] should be a good solution. After the calculation of uncertainty associated to error of indication, the \( E_n \) value is to be calculated for each measurement point, using the actual values and those from the calibration certificate.

4.2. Accredited medical laboratories

The intermediate checks in medical field, should be performed depending on the laboratory’s requirements.

4.2.1. If the instrument is not checked at a constant time, then the next method can be used: a weight of appropriate accuracy class is placed “\( n \)” times (usually three) on the pan, obtaining the respective readings. The average of these indications should be within the range:

\[
\overline{I} \in [M_{\text{ext}} \pm u_c] \tag{7}
\]

where: \( M_{\text{ext}} \) is the conventional mass of the standard weight from the calibration certificate and \( u_c \) is standard uncertainty comprising uncertainty of the weight used and that of the balance in the respective measurement point. It is mentioned that the indications of the balance shall be corrected with the values from the calibrated certificate. If the correction is not applied, its value shall be added to uncertainty of the balance. It is advisable that the intermediate check be repeated for two other loads.
4.2.2 If the balance is daily checked, using the same weight, it can be used either a control chart, Figure 1, either a determination of “Bias”[5], according to Table 2, where:

\[
Bias = 100 \times \frac{\text{Laboratory average value} - \text{Target value}}{\text{Target value}}
\]

This measure must be less than or equal to the specified permissible value from the manufacturer’s specification, if this information is available. If the minimum allowed value of the bias is not specified by the manufacturer, then, the laboratory must establish its own criteria. Also, a standard deviation (SD) is used to evaluate a balance with regard to its reproducibility, improving process performance over time by studying variation, Figure 1. For a confidence interval of ± 3 SD, approximately 99% of the values measured lie within these limits around the mean. An example is presented in Table 2.

| No. of IC | Measured values | Average | SD | Bias % | Target Value | -1SD | +1SD | -2SD | +2SD | -3SD | +3SD |
|----------|-----------------|---------|----|--------|--------------|------|------|------|------|------|------|
|          | 10.065, 10.057, 10.058, 10.065 | 10.065 | 0.010 | 0.001 | 10.065 | 10.058 | 10.072 | 10.051 | 10.079 | 10.045 | 10.086 |

Figure 1. The evaluation of the balance using a control chart

5. Conclusions
According to [1], one of the methods used for ensuring the validity of results is the use of intermediate checks. The paper presents different approaches regarding intermediate checks performed in the field of mass measurements: in the calibration/verification of weights, of mass comparators and NAWI. For each measurement instrument is presented an acceptance criterion. If the acceptance criterion is exceeded during intermediate check, the laboratory should take proper corrective actions. These control procedures are important either to decide whether the recalibration interval can be maintained, prolonged, reduced or to take other appropriate corrective actions.

6. References
[1] ISO/IEC 17025/2017 General requirements for the competence of testing and calibration laboratories
[2] ISO 13528/2015 Statistical methods for use in proficiency testing by inter-laboratory comparison
[3] OIML R111 Weights of classes E1, E2, F1, F2, M1, M1-2, M2, M2-3 and M3
[4] Euramet Calibration Guide, cg-18, v.4/2015 Guidelines on the Calibration of Non-Automatic Weighing Instruments
[5] EURACHEM/CITAC guide cg 4/2012, Quantifying Uncertainty in Analytical Measurement