# 6.2 Technical Criteria for Mass Laboratories<sup>1</sup>

## 6.2.1 Scope

The purpose of this section is to specify the specific technical criteria needed to meaningfully assess the competence of a calibration laboratory that performs mass calibrations.

**6.2.1.1** A laboratory should declare its measurement capability in terms of uncertainties for each mass value. For many laboratories these uncertainties correspond to three arbitrary echelons of measurements that roughly correspond to weight classifications at nominal mass value ranges of measurements. For laboratories seeking Recognition at an uncertainty range that corresponds to a specific echelon, the scope of Recognition should follow the echelons as defined in Table 4 with a declared range of nominal mass values and their associated uncertainties. Recommended ranges of mass values are provided in Table 5. A summary of sections 6.2.3, 6.2.5.1, 6.2.5.6, and 6.2.6.1 are provided in Table 8.

**6.2.1.2** The echelon stated by the laboratory is associated with the standards, procedures, measurement control, facilities, equipment, staff capability, and the overall level of performance according to Table 4, and is specifically defined by the expanded uncertainty. However, echelons are evaluated by all of the factors in Table 8 in addition to the laboratory's reported uncertainties.

Echelon	Verification Levels	Expanded Uncertainty of the Measurements	
I, (Extra Fine Accuracy)	OIML Classes E <sub>1</sub> , E <sub>2</sub> ASTM Classes 0, 1		
II, (Fine Accuracy)	OIML Classes F <sub>1</sub> , F <sub>2</sub> ASTM Classes 2, 3	The expanded uncertainty must be less than 1/3 of stated tolerances at all levels.	
III, (Medium Accuracy)	OIML Classes M <sub>1</sub> , M <sub>1-2</sub> , M <sub>2</sub> , M <sub>2-3</sub> , M <sub>3</sub> ASTM Classes 4, 5, 6, 7 NIST Class F		

Table 4.Mass calibration echelons

<sup>&</sup>lt;sup>1</sup> This section was originally developed by WMD for adoption in the NVLAP Calibration Laboratories Technical Guide (NIST HB 150-2G); it is modified here for WMD application.

Echelon	<b>Ranges of Recognition</b> <b>Nominal Value Ranges</b>		
	$\geq$ 30 kg (define limit)		
Ι	30 kg to 1 mg		
	1 kg to 1 mg		
	100 g to 1 mg		
II	$\geq$ 30 kg (define limit)	for special applications:	
	30 kg to 1 mg	$\geq$ 1000 lb (define limit)	
	1 kg to 1 mg	$\geq$ 50 lb (define limit)	
	100 g to 1 mg	50 lb to 0.001 lb	
III	$\geq$ 30 kg (define limit)	normal applications:	
	30 kg to 1 g	$\geq$ 1000 lb (define limit)	
	1 kg to 1 g	$\geq$ 50 lb (define limit)	
	100 g to 1 g	50 lb to 0.001 lb	

Table 5. Typical "Ranges of Recognition" for mass calibration

**6.2.1.3** The reported uncertainty of mass standards calibrated by a mass calibration laboratory will vary depending on available balances, the uncertainty of reference standards, and the nominal value of the mass standard being tested. Thus, a laboratory may perform calibrations at Echelon I in some ranges, for example at 1 kg, and may perform calibrations at Echelon II, e.g., 20 kg, in other ranges. The laboratory performs calibrations in a specified range as requested; however, all laboratories may not be capable of meeting the requirements of all echelons. Differing equipment, skills, knowledge, measurement control, and demonstrated competence are required for each of the echelons. Demonstrated competence in one echelon is not sufficient to guarantee adequate performance in the others.

#### 6.2.2 References

- [1] ANSI/ASTM E 617-97, Standard Specification for Laboratory Weights and Precision Mass Standards, March 1998.
- [2] OIML R 111-1, Weights of classes E1, E2, F1, F2, M1, M1–2, M2, M2–3 and M3 Part 1: Metrological and technical requirements, 2004.
- [3] NIST Handbook 105-1, 105-8, Specifications and Tolerances for Reference Standards and Field Standard Weights and Measures, Specifications and Tolerances for Field Standard Weights, (NIST Class F), 1990, and Field Standard Weight Carts, 2003.
- [4] NBS Technical Note 844, Designs for the Calibration of Small Groups of Standards in the Presence of Drift, Cameron, J. M., Hailes, G. E., 1974.
- [5] NBS Technical Note 952, Designs for the Calibration of Standards of Mass, Cameron, J. M., Croarkin, M. C., Raybold, R. C., 1977.
- [6] NISTIR 5672, Advanced Mass Calibrations and Measurement Assurance Program Requirements for State Calibration Laboratories, Fraley, K. L., Harris, G. L., 2005.
- [7] NISTIR 6969, Selected Laboratory and Measurement Practices, and Procedures, to Support Basic Mass Calibrations, Harris, G. L., Torres, J. A., 2003.

#### 6.2.3 Statistical process control

**6.2.3.1** Appropriate measurement assurance programs should be in place and available for review for each echelon and nominal mass range for which calibration data is provided. Note Table 8 for appropriate measurement control programs for each echelon. Appropriate data includes balance standard deviations that represent process variation and well-characterized check standard values.

**6.2.3.2** Measurement assurance techniques should exhibit results consistent with the procedures used to perform calibrations and should be integral to the measurement to accurately reflect the measurement process. For those situations where statistical information is not inherent to the process, i.e., simple measurements without built-in redundancy checks, additional measurements should be made to provide experimental characterization of the measurement sufficient for an adequate estimation of the process uncertainty. Those data should be available for review.

#### 6.2.4 Accommodation and environment

**6.2.4.1** To be deemed capable of making adequate measurements, calibration laboratories should provide an environment with adequate environmental controls appropriate for the level of measurements to be made, according to echelons defined herein. The environmental conditions are summarized in Table 6.

Echelon	Temperature Limits*	Relative Humidity Limits (maximum change per 4 h)
Ι	20 °C to 23 °C, a set point <u>+</u> 1 °C maximum change 0.5 °C/h	40 % to 60 % <u>+</u> 5 %
П	20 °C to 23 °C, a set point <u>+</u> 2 °C maximum change 1 °C/h	40 % to 60 % <u>+</u> 10 %
III	18 °C to 27 °C, maximum change 1 °C/h	40 % to 60 % <u>+</u> 20 %

 Table 6.
 Environmental facility guidelines for mass laboratories

\*Limits established in this table are intended to provide basic parameters to ensure temperature and humidity stability in the laboratory. While maximum changes are noted, they do not imply rate of change or a net change over time. Environmental changes at these limits within a very short period of time will adversely affect mass measurements.

**6.2.4.2** Environmental conditions must be within the specifications of the weighing instruments where applicable. Environmental deviations from those specified must address possible measurement impacts, identify appropriate laboratory procedures that are used to minimize measurement errors, and have components incorporated into measurement uncertainties. Deviations for temperature and relative humidity controls are not necessarily corrected by performing buoyancy corrections and may adversely affect measurement results. Errors due to thermal convection currents or changes to moisture layers on the surface of mass standards may be significant when the environment is not controlled to within these limits and/or when artifacts are not allowed to equilibrate properly.

**6.2.4.3** Cleanliness guidelines are usually met without clean-room type air handling systems by maintaining clean-room type practices with suitable filtration. The laboratory must maintain limited access to the calibration area and minimize contamination (provide a clean surface) for locations where calibration items are being tested. Activities such as smoking, eating, or drinking and items such as paper products, printers, and files contribute to the difficulty of maintaining adequate cleanliness and are not recommended. Excessive air exchange rates negatively affect balance performance; therefore, air flow is generally minimized to the extent possible. A positive pressure, laminar-type air flow is usually needed to maintain cleanliness recommendations and to minimize air currents.

**6.2.4.4** Vibration should not diminish the performance of precision analytical balances and mass comparators. Proximity to heavy machinery, railways, heavily traveled highways, or similar sources of known vibration is not recommended. Steps are often taken to attenuate vibration to an acceptable level of stability with methods such as massive piers (solid marble or concrete tables), isolated foundations, or elimination of the source. Balances and

mass comparators used for Echelons I and II generally require massive piers, independent piers, and/or an isolated foundation; pneumatic or hydraulic tables are inappropriate.

**6.2.4.5** Undesirable effects due to static electricity should be controlled, if needed, with methods such as humidity, antistatic deionizing radiation devices, the grounding of balances or operators, or with the use of special conductive flooring and selection of proper clothing for staff.

### 6.2.5 Equipment and reference materials

**6.2.5.1** Minimum reference standards should be available at each echelon and range, for which the laboratory is Recognized, as recommended in Table 8. Sufficient historical data and uncertainty analysis should be available to support the standards used.

**6.2.5.2** The accuracy of auxiliary instruments for Echelons I and II, (e.g., scale, analytical balance, and mass comparator) is less important than the precision of the instrument due to algorithms used in mass calibration. However if such equipment is repaired, it should be reevaluated to ascertain its current level of precision prior to use, and the uncertainty estimate should reflect the post repair performance.

**6.2.5.3** The precision of the scale, analytical balance, or mass comparator, as determined through appropriate process control charts, should be suitable for the echelon at which it is used. For an application where external standards are used for comparison, appropriate control charts should be maintained to evaluate the process standard deviation. Note Table 8 summary for further evaluation.

**6.2.5.4** Means should be provided to measure barometric air pressure, air temperature, and relative humidity of the laboratory environment as indicated in Table 7; documentation of the accuracy and traceability is required. These instruments should be used in close proximity to the balance being used. For Echelon I, temperature may be measured inside the weighing chamber when there is a difference between the air temperature in the balance chamber and the surrounding area. For Echelon III, where buoyancy corrections are generally negligible, recording environmental data provides evidence to support compliance with general environmental requirements of the previous section but the accuracy generally does not affect measurement results when appropriate artifact equilibration occurs.

Parameter	<b>Barometric Pressure</b>	Temperature	<b>Relative Humidity</b>
Echelon I	± 65 Pa (0.5 mm Hg)	$\pm 0.1$ °C	± 5 %
Echelon II	± 135 Pa (1.0 mm Hg)	$\pm 0.5$ °C	$\pm$ 10 %
Echelon III	The laboratory maintains documented accuracy.		

 Table 7. Environmental equipment accuracy (expanded uncertainty)

**6.2.5.5** For Echelon I, the laboratory should state the presence of a possible systematic error in the combined uncertainty associated with the use of an assumed density in the primary or reference standards (additional Type B component) or the laboratory should have appropriate means to measure the density of mass standards. If the magnetic susceptibility of the mass standards is evaluated, it should be indicated on calibration reports. The methods used to determine density or magnetic susceptibility should be documented.

**6.2.5.6** Each mass standard used as a reference standard by the laboratory should be calibrated by NIST or by an accredited or Recognized laboratory with capability adequate to sustain the accuracy required and maintain traceability to the SI. The laboratory should provide evidence, such as periodic surveillance, that the standard is, in principle, acceptable for providing calibration services at each echelon. Note Table 8 for traceability guidelines.

**6.2.5.7** Balances used as a direct comparison to the mass unit, should be given a verification test or calibration prior to use. For an application requiring balance accuracy, the laboratory should choose appropriate and correct calibration algorithms. Balances used as dividers and multipliers of the mass unit should be capable of the appropriate accuracy and linearity requirements of the accuracy class for which they are used. Calibration of built-

in standards should be performed periodically and should be verified prior to use. History from measurement control programs (surveillance testing) may be used to determine calibration intervals.

Instruments used to monitor environmental conditions in the laboratory should be traceable to a suitable national laboratory (directly or via an accredited or Recognized laboratory) and be recalibrated periodically unless defining standards are employed. Calibration periods will be documented by the laboratory.

**6.2.5.7** Balances used as a direct comparison to the mass unit, should be given a verification test or calibration prior to use. For an application requiring balance accuracy, the laboratory should choose appropriate and correct calibration algorithms. Balances used as dividers and multipliers of the mass unit should be capable of the appropriate accuracy and linearity requirements of the accuracy class for which they are used. Calibration of built-in standards should be performed periodically and should be verified prior to use. History from measurement control programs (surveillance testing) may be used to determine calibration intervals.

**6.2.5.8** Instruments used to monitor environmental conditions in the laboratory should be traceable to a suitable national laboratory (directly or via an accredited or Recognized laboratory) and be recalibrated periodically unless defining standards are employed. Calibration periods will be documented by the laboratory.

#### 6.2.6 Calibration methods

**6.2.6.1** The algorithm chosen for the measurement, the reference standard to be used, and the equipment to be used for a particular calibration should provide acceptable levels of uncertainty for that calibration. A documented procedure should be available in the laboratory to determine the correct algorithm. Note Table 8 for guidelines.

**6.2.6.2** Computer programs should have passed software quality analysis. Computer programs should be tested, using standard data sets designed to magnify errors, as an effective way of showing that program errors are not present which do not affect some measurements but cause others to be incorrect. Computer programs should be documented in detail. The documentation should include technical references that provide the basis for the algorithm, the weighing equation, and the data set used to test the program for errors.

#### 6.2.7 Handling of calibration items

**6.2.7.1** The laboratory should have documented procedures to ensure adequate chain-of-custody of calibration items if required by law.

**6.2.7.2** The laboratory will document appropriate procedures to ensure that cleaning, if performed, ensures the integrity of the standards, and to provide for thermal conditioning, where appropriate.

**6.2.7.3** The laboratory must allow adequate stabilization time for mass standards to ensure environmental and thermal stability prior to calibration.

**6.2.7.4** Documented procedures to ensure adequate tracking of calibration items should be appropriate to the class of mass standard. Strings, tags, or labels fastened to the standard are inappropriate.

#### 6.2.8 Calibration certificates and test reports

**6.2.8.1** In addition to meeting the criteria in section 5.10 of this handbook, calibration certificates and test reports should describe the mass standards mentioned in the report with sufficient detail to avoid any ambiguity. For Echelons I and II, additional items to be included on a test report, are: mass (true mass) values, conventional (apparent) mass values, reference density, uncertainties, material, thermal coefficient of expansion (if used in calculations), construction, density (measured or assumed), and any identifying markings.

**6.2.8.2** Environmental parameters measured during the test should be provided on certificates and reports for Echelons I and II. Typical ranges are acceptable for reporting conditions for Echelon III. These include laboratory temperature, barometric pressure and relative humidity.

**6.2.8.3** Information regarding cleaning methods (if requested) should be provided on the test report.

**6.2.8.4** Reports may include reference to NIST, OIML or ASTM classification schemes and tolerances. Calibration items being tested should meet appropriate specifications for evaluation as well as tolerances. It is the responsibility of the requestor of the calibration to select classifications acceptable for their needs. In the case where magnetism, surface finish, and density are not tested for Echelon I and II, a statement to that effect should be included on the calibration report.

**6.2.8.5** The weight surface of a mass standard should be free of any sign of abuse or damage. Signs of abuse or misuse include the placement of labels, tags, wires or other material on mass standards. In addition, visible dirt and fingerprints are signs of misuse for Echelons I and II. It is recommended that the calibration laboratory establish appropriate means for notifying customers regarding any unusual factors such as signs of abuse regarding the mass standard being tested should be included on the report. Out of tolerance conditions should be reported when significant or when requested.

Echelon	Minimum Measurement Assurance (5.9)	Minimum Reference Standards (5.6)	Minimum Traceability (5.6)	Minimum Calibration Methods (5.4)
I (Extra Fine)	<ul> <li>Process control charts</li> <li>Check standards for each decade, with long term standard deviation</li> <li>Surveillance of all standards</li> <li>Proficiency Testing -on-site assessment -round robin participation</li> </ul>	<ul> <li>OIML Class E<sub>1</sub>, or E<sub>2</sub></li> <li>Or</li> <li>ASTM Class 0, 1</li> <li>single piece, highly polished</li> </ul>	<ul> <li>NIST, or other national level calibration every 2 to 5 years based on measurement process data and independent verification, and</li> <li>Suitable surveillance testing and participation in PTs at this level</li> <li>Documented with GMP 11 and 13</li> </ul>	<ul> <li>Documented weighing designs consisting of redundant comparisons, with built-in process controls such as those used at the national level.</li> <li>For example:</li> <li>Technical Notes, 952 and 844, SOP 5, SOP 28</li> </ul>
II (Fine)	<ul> <li>Process control charts</li> <li>Check standards for each decade, with long term standard deviation, per SOP 9</li> <li>Surveillance of selected standards Proficiency Testing</li> <li>-on-site assessment</li> <li>-round robin participation</li> </ul>	<ul> <li>OIML Class E<sub>1</sub>, or E<sub>2</sub></li> <li>ASTM Class 0, 1; or</li> <li>single piece, highly polished</li> </ul>	<ul> <li>NIST calibration every 2 to 5 years based on measurement process data and independent verification, or</li> <li>Calibration by accredited or Recognized Echelon I laboratory, if uncertainty requirements can be met.</li> <li>Documented with GMP 11 and 13</li> </ul>	<ul> <li>Documented comparison calibration procedure</li> <li>For example:</li> <li>NISTIR 6969, SOP 4, SOP 5, or equivalent validated procedure</li> </ul>
III (Medium)	<ul> <li>Process control charts</li> <li>Check standards for each balance per SOP 9</li> <li>Proficiency Testing -on-site assessment -round robin participation</li> </ul>	<ul> <li>Working standards such as:</li> <li>ASTM Class 2, 3 Or</li> <li>OIML Class F1</li> <li>two piece acceptable, fine finish</li> </ul>	<ul> <li>Calibration of all working standards by NIST every 2 to 5 years based on measurement process data and independent verification, or</li> <li>Calibration of all working standards by an Echelon I or II accredited or Recognized labs every 2 to 5 years based on measurement process data and independent verification</li> <li>Documented with GMP 11 and 13</li> </ul>	<ul> <li>Use of annually calibrated balance with documented and validated verification procedure prior to each use</li> <li>NISTIR 6969, SOP 8, SOP 7</li> <li>Other documented and verified procedure</li> </ul>

Table 8. Summary of technical criteria for mass calibration