

D3 Volume calibrations

D3.1 General

The purpose of this section is to provide the specific technical requirements to assess the competence of a calibration laboratory that performs volume calibrations. Volumetric measurements are obtained from mass measurements of known-density materials. Volume calibrations may be determined by either a gravimetric (weighing procedure) or a volume transfer (comparative) procedure. The two types of procedures have different technical requirements, and both are defined here. Note that the type of calibration procedure used affects the achievable uncertainty.

D3.2 Accommodations and environmental conditions

D3.2.1 Vibration, air currents, rapid temperature fluctuations, and other environmental variations shall be kept to levels such that they do not diminish the validity of the measurement whether by volume transfer methods or the performance of precision balances or scales when gravimetric methods are used.

D3.2.2 Relative humidity shall be monitored more closely when evaporation or condensation may be a concern.

D3.3 Calibration methods and method validation

D3.3.1 The algorithm chosen for the measurement, the reference standard to be used, and the equipment to be used for a calibration shall be correct for that calibration.

D3.3.2 A documented procedure shall be available in the laboratory.

D3.4 Equipment, standards and reference materials

D3.4.1 Gravimetric standards and equipment

D3.4.1.1 Mass standards used as reference standards shall be traceable to the International System of Units through standards maintained by a national metrology institute (such as NIST) and be available at each class and range for which the laboratory is accredited.

D3.4.1.2 When water is used as the medium for gravimetric methods, it shall be deionized or distilled, and its density shall be verified.

D3.4.1.3 For gravimetric procedures the density shall be calculated/measured to 0.000001 g/cm^3 .

D3.4.1.4 The quality of water used as a calibration medium shall be of adequate purity (potable) and cleanliness and free from excess air entrapment.

D3.4.1.5 Gravimetric methods shall be performed using weighing equipment with adequate accuracy and precision for the uncertainty of the measurement procedure.

D3.4.1.6 Appropriate control charts or range charts shall be maintained to verify the volume measurement process.

D3.4.1.7 Mass calibration variability shall not be used to estimate variability for gravimetric volume calibrations.

D3.4.1.8 Gravimetric methods require the means to adequately measure barometric air pressure, air temperature, water temperature, and relative humidity of the laboratory environment to perform proper

buoyancy corrections and calculate or look up accurate water density. Environmental measuring equipment shall be appropriate to support the volume calibration method used.

D3.4.2 Volumetric standards

D3.4.2.1 Volume standards used as reference standards in the laboratory shall be traceable to the SI through standards maintained by a national metrology institute (such as NIST).

D3.4.2.2 The laboratory shall have appropriate programs and procedures in place for verification and recalibration of its volume standards.

D3.4.2.3 Volumetric methods require accurate temperature measurements. Environmental measuring equipment shall be appropriate to support the volume calibration method used.

D3.4.2.4 Extreme care shall be used to measure water temperatures. The accuracy of water density calculations is severely degraded by inaccurate water temperature measurements and the presence of thermal gradients.

D3.5 Handling of calibration items

D3.5.1 The volume standard being calibrated shall be free of any sign of abuse or damage, such as dents, chips, improper draining due to lack of cleanliness, and dirty sight gages.

D3.5.2 The laboratory shall have documented procedures to ensure adequate chain-of-custody of calibration items if required by law.

D3.5.3 Procedures shall be documented to ensure adequate tracking glass or metal volumetric standards.

D3.6 Assuring the quality of calibration results

D3.6.1 Measurement control programs shall be in place and available for review for each measurement type (based upon procedures) and nominal volume range for which calibration data is provided.

D3.6.2 Measurement control techniques shall be implemented, with the resulting data available for review.

D3.6.3 For those situations in which statistical information is not inherent to the process, i.e., simple measurements without built-in redundancy checks, additional measurements shall be made to provide experimental characterization of the measurement that is sufficient for an adequate estimation of the measurement uncertainty.

D3.7 Reporting the results

D3.7.1 As required by ISO/IEC 17025, calibration reports shall describe the volume standards with sufficient detail to avoid any ambiguity. In addition to those items required by ISO/IEC 17025, calibration reports shall contain the following items:

- a) volume;
- b) uncertainty;
- c) reference temperature;

- d) material of the standard submitted for calibration;
- e) thermal coefficient of expansion (assumed, measured, or provided by the manufacturer) used;
- f) construction of the item being calibrated;
- g) any identifying markings;
- h) any tolerances if appropriate (See section regarding conformity statements).

D3.7.2 Environmental parameters measured during the calibration shall be provided on the calibration report as appropriate. These measurements include laboratory air temperature, volume standard temperature, temperature of the medium, barometric pressure, and relative humidity.

D3.7.3 Conformity assessment to a classification scheme is often required for customers using volume standards. If a statement of conformity is being made:

- a) the volume standards being calibrated shall meet the appropriate classifications such as NIST, ASTM, API, or OIML;
- b) the calibration reports shall clearly identify the classification scheme with revision date to which the conformity assessment was made;
- c) conformity statements shall clearly specify to which results they apply and which specifications and tolerances, or parts thereof, are met or not met;
- d) out-of-tolerance conditions shall be reported.