6.3 Technical Criteria for Volume Laboratories[1]

6.3.1 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 volume calibrations. It should be noted that the procedures affect the achievable uncertainty.

Table 9. Typical "Ranges of Recognition" for volumetric calibration

Procedure Types & Echelon	**Scope of Recognition Nominal Value Range**		
Echelon I Gravimetric	Glassware		Metal Test Measures or Provers
	Standard	Pipettes & Syringes	
	≥ 1 L		≥ 2 000 L **or** ≥ 500 gal
	1 L to 100 mL		100 L < V < 2 000 L **or** 25 < V < 500 gal
	100 mL to 1 µL	100 mL to 1 µL	≤ 100 L **or** ≤ 25 gal
Echelon II Volume Transfer	Glassware		Metal Test Measures or Provers
	≥ 1 L **or** 1 qt		≥ 2 000 L **or** ≥ 500 gal
	100 mL < V < 1 L **or** 1 gill < V < 1 qt		100 L < V < 2 000 L **or** 25 < V < 500 gal
	…		≤ 100 L **or** ≤ 25 gal

6.3.1.1 Volumetric units are derived from mass units. Volume calibrations may be determined by either a gravimetric (weighing procedure) or a volume transfer (comparative) method. The two methods require different technical requirements and both are defined here. The measurement of volume by metering methods (and meter calibration) is outside the scope of this document.

6.3.1.2 The gravimetric procedure is based on the conservation of mass principle where a determination of the mass of water contained in or delivered from the vessel that is being calibrated is used to define volume. The mass values are determined in air, are corrected for air buoyancy effects, and are corrected to appropriate reference temperatures. The accuracy and precision will vary depending on balances used, the purity of the water, the ability to make accurate temperature measurements, the nominal value of the volume standard being tested, and the ability to make adequate mass measurements.

6.3.1.3 In the volume transfer procedure, water is delivered from a primary volume standard to the vessel under test. Temperature corrections are made to compensate for the cubical coefficients of thermal expansion of the standard, test vessel, and water to a specified reference temperature. The accuracy and precision will vary considerably depending on the presence of a meniscus, the cleanliness and drain characteristics of the container, the cleanliness and purity of the water, and the ability to make adequate temperature measurements.

6.3.2 References

[1] ANSI/ASTM E 287-02, Standard Specification for Laboratory Glass Graduated Burets (2002).

[1] This section was originally developed by WMD for adoption in the NVLAP Calibration Laboratories Draft Technical Guide (NIST HB 150-2G); it is modified here for WMD application.

[2] ANSI/ASTM E 288-06, Standard Specification for Laboratory Glass Volumetric Flasks (2006).

[3] ANSI/ASTM E 438-92 e1, Standard Specification for Glasses in Laboratory Apparatus (2006).

[4] ANSI/ASTM E 542-01, Standard Practice for Calibration of Volumetric Apparatus (2001).

[5] ANSI/ASTM E 694-99, Standard Specification for Laboratory Glass Volumetric Apparatus (2005).

[6] ANSI/ASTM E 969-02, Standard Specification for Glass Volumetric (Transfer) Pipets (2002).

[7] OIML R 4, Volumetric Flasks (one mark) in Glass (1972).

[8] OIML R 40, Graduated Pipettes for Verification Officers (1981).

[9] OIML R 41, Standard Burettes for Verification Officers (1981).

[10] OIML R 43, Standard Graduated Glass Flasks for Verification Officers (1981).

[11] NIST Handbook 105-2, 105-3, 105-4, 105-7 Specifications and Tolerances for Reference Standards and Field Standard Weights and Measures; Specifications and Tolerances for Field Standard Glass Flasks, Open-neck Provers, LPG Volumetric Provers, Dynamic Small Volume Provers.

[12] API Manual of Petroleum Measurement Standards, Chapter 4 - Proving Systems; Section 3, Small Volume Provers, 1988; Section 4, Tank Provers, 1998 (Reaffirmed November 2005); Section 7, Field Standard Test Measures, 1998 (Reaffirmed October 2003).

[13] NISTIR 7383, Selected Procedures for Volumetric Calibrations, November 2006.

[14] NIST Special Publication, 250-72, NIST Calibration Services for Liquid Volume, March 2006.

6.3.3 Statistical process control

6.3.3.1 Appropriate measurement control programs should be in place and available for review for each measurement type (based on procedures) and nominal volume range for which calibration data is provided. Note Table 12 for appropriate measurement control programs for each measurement type. Appropriate data includes standard deviations and range values that represent process variation and well characterized check standard values.

6.3.3.2 Measurement control techniques should exhibit results consistent with the procedures used to perform calibrations and should be integrated into 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.

6.3.4 Accommodation and environment

6.3.4.1 To be deemed capable of making adequate measurements, calibration laboratories should provide a facility with adequate environmental controls appropriate for the level of measurements to be made, according to procedure types as shown in Table 10. Lower relative humidity may increase measurement error due to evaporation.

Table 10. Environmental facility guidelines for volumetric calibration

Echelon & Procedure	Temperature	Relative Humidity (maximum range per 4 hours)
I Gravimetric	20 °C to 23 °C, set point ± 2 °C maximum change 1.0 °C/h	40 % to 60 % ± 10 %
II Volume Transfer	18 °C to 27 °C maximum change 2.0 °C/h	40 % to 60 % ± 20 %

NOTE: The environmental conditions should also be within the specifications of applicable equipment. Gravimetric calibrations must comply with the comparable sections of 6.2 for mass calibrations because this procedure is a high-precision mass calibration with additional complexities.

6.3.4.2 The environment in which testing activities are undertaken should not invalidate the results or adversely affect the required accuracy. Particular care should be taken when such calibration practices are undertaken at sites other than the permanent laboratory facility to minimize the effects of uncontrolled environments (e.g., outside or in open bays.)

6.3.4.3 Vibration, air currents, rapid temperature fluctuations, and other environmental concerns should not diminish the accuracy and precision of volume transfer methods or the performance of precision balances or scales when gravimetric methods are used.

6.3.4.4 The quality of water used as a calibration medium should be of adequate purity (potable) and cleanliness, and should be free from excess air entrapment. For gravimetric procedures the density should be calculated/measured to the nearest 0.00001 g/cm^3.

6.3.5 Equipment and reference materials

6.3.5.1 Mass standards used as reference standards should be traceable to a national laboratory (such as NIST) and be available at each class and range, for which the laboratory is Recognized, as recommended in Table 12. Sufficient historical data and uncertainty analysis should be available to support the standards used.

6.3.5.2 Volume standards used as reference standards in the laboratory should be traceable to a national laboratory (such as NIST) and the laboratory should have appropriate procedures in place for verification and recalibration. The accuracy of the primary volume standards of the laboratory should be appropriate for the accuracy class of services provided.

6.3.5.3 Gravimetric methods, which generally use water as calibration media, require the verification of an adequate supply of deionized or distilled water.

6.3.5.4 Gravimetric methods require the use of weighing equipment with adequate accuracy and precision for the uncertainty of the measurement procedure. Appropriate control charts or range charts should be maintained to verify the measurement process.

6.3.5.5 Gravimetric methods require the means to adequately measure barometric air pressure, air temperature, water temperature, and relative humidity of the laboratory environment. Volumetric methods require temperature measurements. Environmental measuring equipment should be available with the accuracy indicated in Table 11. Relative humidity may need to be monitored more closely if evaporation is a concern.

Table 11. Environmental equipment accuracy (expanded uncertainty)

Procedure Type	Barometric Pressure	Temperature water/air	Relative Humidity
Gravimetric	± 135 Pa (1.0 mm Hg)	± 0.1 °C / ± 0.5 °C	± 10 %
Volume Transfer	Not essential	± 0.5 °C	Not essential

6.3.6 Calibration methods

6.3.6.1 The algorithm chosen for the measurement, the reference standard to be used, and the equipment to be used for a particular calibration should be correct for that calibration. A documented procedure should be available in the laboratory to determine the correct algorithm. (Examples are provided in NIST Handbook 145 Standard Operating Procedures, SOPs.)

6.3.6.2 Computer programs should have passed software quality analysis. Computer programs should be documented in detail. The documentation should include technical references that provided the basis for the algorithm, any weighing equations, and data set used to test the program for errors.

6.3.7 Handling of calibration items

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

6.3.7.2 Appropriate procedures should be documented to ensure adequate tracking of calibration items that are appropriate for glass or metal volumetric standards.

6.3.8 Calibration certificates and test reports

6.3.8.1 In addition to meeting the criteria in section 5.10 of this handbook, calibration certificates and test reports should describe the volume standards mentioned in the report with sufficient detail to avoid any ambiguity. Additional items to be included on a test report are: volume, uncertainty, reference temperature, material, thermal coefficient of expansion (assumed or measured), construction, any identifying markings, and any tolerances if appropriate.

Environmental parameters measured during the test should be provided on the test report as appropriate. These include laboratory temperature, volume standard temperature, barometric pressure and relative humidity.

6.3.8.3 Volume standards being tested should meet the appropriate specifications such as NIST, ASTM, API, or OIML, if required by laboratory customers. It is the responsibility of laboratory customers to determine acceptable accuracy levels for their needs.

6.3.8.4 The calibration item (volume standard) should be free of any sign of abuse or damage. Signs of abuse or misuse include dents, chips, improper draining due to lack of cleanliness, and dirty sight gages. Out of tolerance conditions should be reported.

Table 12. Summary of technical criteria for volumetric calibration

Echelon	Minimum Measurement Control	Minimum Reference Standard	Minimum Traceability	Minimum Calibration Methods
I Gravimetric	• Process control charts • Check standards • Surveillance of all standards used to provide measurement services • Proficiency testing - On-site assessment - Round robin participation	• Appropriate mass standards - ASTM Class 2 or 3; or - OIML Class F_1 or F_2; or - Calibrated Balance	• NIST, or national level calibration periodically, based on independent historical data verification or • - Test of weighing equipment using correct methods of calibration and adjustment with traceable mass standards	• Documented comparison calibration procedure or • NISTIR 7383, SOP 14 (for example) or • Use of calibrated balance
II Volume Transfer	• Process control charts • Check standards • Surveillance of selected standards • Proficiency testing - On-site assessment - Round robin participation	Primary volume standards with accuracy and repeatability characteristics acceptable for the type of service provided	• Original NIST calibration and periodic independent verification or • Calibration by accredited or Recognized laboratory, if uncertainty requirements can be met	Documented volume transfer (or water draw) procedure recognized by NIST, OIML, ASTM, or API