



Designation: E542 – 22

Standard Practice for Gravimetric Calibration of Laboratory Volumetric Instruments¹

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1. Scope

1.1 This practice covers procedures for use in the calibration of volumetric instruments that include glassware, plasticware, and laboratory standards that are in common use in chemical, analytical, clinical, and calibration laboratories. It is based on the gravimetric determination of the quantity of pure water, either contained or delivered at a calibration temperature, and the conversion of this value to a volume at a given reference temperature, normally 20 °C by means of suitable equations. Calibration using mercury is excluded. Calibration may be performed using alternative gravimetric methodology, if it is demonstrated and documented that the results obtained are equivalent to those obtained using the methodology described herein. Alternative reference temperatures and associated equations are provided.

1.2 This practice is intended to encompass volume capacity instruments between the limits of 0.1 cm³ and 10 000 cm³. Typical volumetric instruments falling within the purview of this practice are burettes graduated “to deliver,” graduated cylinders, volumetric flasks, measuring and dilution pipettes, transfer and capacity pipettes such as those in Specification E694, specific gravity flasks such as those used in several ASTM standards, and metallic volumetric standards such as those used in legal metrology.

1.3 The procedures are not recommended for calibration of volumetric instruments with capacities below 0.1 cm³, such as microglassware without incorporating evaporation corrections; evaporation methods and corrections are not provided. Capacities given in 1.2 are not intended to be maximum capacity limitations; volumes greater than 10 000 cm³ may be calibrated with this procedure. Maximum capacity limitations are based on available equipment, standards, adequate quantities of pure water, and the ability to safely handle large volumetric instruments.

¹ This practice is under the jurisdiction of ASTM Committee E41 on Laboratory Apparatus and is the direct responsibility of Subcommittee E41.01 on Laboratory Ware and Supplies.

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1.4 This standard may be used for the calibration of volumetric instruments made from materials of glass, plastic, various stable metals, or any other stable materials provided appropriate volumetric coefficients of expansions are available.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

- D1193 Specification for Reagent Water
- E617 Specification for Laboratory Weights and Precision Mass Standards
- E694 Specification for Laboratory Glass Volumetric Apparatus
- E898 Practice for Calibration of Non-Automatic Weighing Instruments

2.2 ISO Standards:³

- ISO 384 Laboratory Glass and Plastics Ware — Principles of Design and Construction of Volumetric Instruments
- ISO 3696:1987 Water for Analytical Laboratory Use. Specification and Test Method
- ISO 4787:2010 Laboratory Glassware – Volumetric Instruments – Methods for Testing of Capacity and for Use
- ISO/IEC 17025:2017 General Requirements for the Competence of Testing and Calibration Laboratories

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

³ Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

2.3 NIST Standards:⁴

- GLP 10** Good Laboratory Practice for the Purity of Water, NISTIR 7383, (2019)
- GLP 13** Good Laboratory Practice for Drying “To Contain” Volume Standards, NISTIR 7383, (2019)
- GMP 3** Good Measurement Practice for Method of Reading a Meniscus Using Water or Other Wetting Liquid, NISTIR 7383, (2019)
- GMP 6** Good Measurement Practice for Cleaning Metal Volumetric Measures, NISTIR 7383, (2019)
- GMP 7** Good Measurement Practice for Cleaning Precision Glassware, NISTIR 7383, (2019)
- GMP 10** Good Measurement Practice for Understanding Factors Affecting Weighing Operations, NISTIR 6969, (2019)
- GMP 11** Good Measurement Practice for Assignment and Adjustment of Calibration Intervals for Laboratory Standards, NISTIR 6969, (2019)
- NIST SP 811** Guide for the Use of the International System of Unit (SI) 2008 Edition
- NIST SP 1038** The International System of Units (SI) — Conversion Factors for General Use (May 2006)
- NISTIR 6969** Selected Laboratory and Measurement Practices to Support Basic Mass Calibrations (2019)
- NISTIR 7383** Selected Procedures for Volumetric Calibrations, (2019)
- SOP 2** Recommended Standard Operating Procedure for Applying Air Buoyancy Corrections, NISTIR 6969, (2019)
- SOP 14** Recommended Standard Operating Procedure for Gravimetric Calibration of Volumetric Standards Using an Electronic Balance, NISTIR 7383, (2019)
- SOP 20** Standard Operating Procedure for Standard Deviation and Range Charts, NISTIR 7383, (2019)
- SOP 29** Standard Operating Procedure for the Assignment of Uncertainty, NISTIR 6969, (2019)

2.4 OIML Standards:⁵

- OIML D 10 (ILAC G-24)** Guidelines for the Determination of Calibration Intervals of Measuring Instruments (2007)⁶
- OIML D 28** Conventional Value of the Result of Weighing in Air (2004)
- OIML R111** Weights of Classes E1, E2, F1, F2, M1, M1—2, M2, M2—3 and M3 Part 1: Metrological and Technical Requirements (2004)

2.5 BIPM Standards:⁷

- GUM: JCGM 100:2008** Evaluation of Measurement Data—Guide to the Expression of Uncertainty in Measurement (Also: ISO/IEC Guide 98-3, same title)

2.6 EURAMET Standards:⁸

- EURAMET/cg-18/V. 4.0** Guidelines on the Calibration of Non-Automatic Weighing Instruments (2015)
- EURAMET/cg-19/V. 3.0** Guidelines on the Determination of Uncertainty in Gravimetric Volume Calibration (2018)
- EURAMET/cg-21/V. 2.0** Guidelines on the Calibration of Standard Capacity Measures Using the Volumetric Method (2020)

3. Summary of Practice

3.1 This practice is based upon a determination of the volume of water either contained in or delivered by a volumetric instrument. Procedures are given for setting the meniscus, handling the instruments, weighing, and converting the weight to the appropriate standard volume.

4. Volumetric Stability and Calibration Frequency

4.1 The primary purpose of this practice is to provide uniform procedures that may be used to accurately calibrate a wide variety of volumetric instruments and volumetric laboratory standards. The techniques are simple in concept and can provide reliable results, provided the procedures are followed consistently. Accordingly, the practice provides a means for checking the original calibration of volumetric instruments and for periodic recalibrations as needed. All volumetric instruments should regularly be inspected prior to use to ensure no visible damage or contamination. Calibration intervals for all volumetric instruments may be based on legal requirements, manufacturer’s recommendations, control charts of similar types of volumetric instruments, analysis of history related to similar models/types of instruments, use and storage frequency as well as the handling conditions and the content of materials being used. References such as the NISTIR 7383, GMP 11, or OIML D 10 (ILAC G-24) may be used to set and adjust calibration intervals.

4.2 The following examples are provided to assist end users:

4.2.1 Under ideal conditions, borosilicate volumetric glassware will hold its calibration indefinitely provided it is used with care and that it is not exposed to hydrofluoric acid, hot phosphoric acid, or strong, hot alkalis, and that it is not heated above 150 °C when dry. A frosting of the glass surface (viewed when dry) indicates that chemical attack has occurred, and recalibration may be in order. As a precaution, it is recommended that glassware always be inspected prior to use to ensure no visible damage or cracks. Significant damage to glassware that impacts the calibration value is likely to be the result of breakage. Volumetric instruments with obvious breaks or cracks should not be used. Glassware should be calibrated at intervals not to exceed five years unless documentation of stability analysis can be provided to support extension of calibration intervals.

4.2.2 Soda-lime volumetric glassware will become frosted with time because of attack from moisture in the atmosphere as well as from the chemicals mentioned above. In addition, it should not be heated above 90 °C when dry. It is

⁴ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, <http://www.nist.gov>.

⁵ Available from Organisation Internationale de Metrologie Legale (OIML), 11 Rue Turgot, 75009 Paris, France, <http://www.oiml.org>.

⁶ Also available from the International Laboratory Accreditation Cooperation, ILAC Secretariat, PO Box 7507, Silverwater NSW 2128, Australia, <https://ilac.org>.

⁷ Available from Bureau International des Poids et Mesures (BIPM), Pavillon de Breteuil, F-92312, Sèvres Cedex, France, <http://www.bipm.org>.

⁸ Available from EURAMET, Bundesallee 100, 38116 Braunschweig, Germany, <http://www.euramet.org>.

recommended, therefore, that it be inspected prior to use for damage or cracks and calibrated at intervals not to exceed five years.

4.2.3 Stainless steel and other metals used in volumetric instruments may be subject to corrosion over time and some may contain mixed materials, such as when they include glass gauge tubes with associated fasteners, where contact points impact the long-term stability. Storage and transport methods may be critical because a minor dent might not be visible on a metal instrument, yet may impact the calibrated volume. Materials other than water (for example, petroleum, metered chemicals) may also cause a chemical reaction with the material and impact the stability of the volumetric instrument over time. Stainless steel volumetric instruments should be inspected prior to each use for damage and contamination and be calibrated at intervals not to exceed one year.

5. Units of Measurement

5.1 *Volume*—The basic SI unit for volume is the cubic meter, m³. Due to its large size, the cubic meter is rarely used in reporting glassware volumetric calibration results but may be appropriate for metallic standards. The cubic centimeter, cm³, is most often used and is employed in this practice. The unit, milliliter, mL, may be considered as equivalent to the cubic centimeter. See NIST SP 811 and NIST SP 1038 regarding conversions and presentation methods for units and symbols.

5.2 *Reference Temperature*—Volumetric instruments for measuring aqueous solutions or liquids other than fuels, and those referenced in Specification E694 are usually calibrated at 20 °C. The procedures described provide for such a calibration. When it is necessary to work at other reference temperatures, such as 27 °C in tropical countries, or for non-laboratory applications, generic equations are provided in Section 15. Possible alternative reference temperatures and applications are provided in Table 1.

6. Equipment and Standards

6.1 *Analytical Balance*—Balances must have enough capacity to weigh the loaded vessel or transfer vessel plus a full quantity of water. The physical dimensions of the balance must also accept the size of the vessels which need to be weighed. The resolution of the balance may be a limiting factor in the accuracy of the measurements. Calibration and uncertainty analysis of the balance may be conducted using Practice E898

or equivalent procedures such as EURAMET/cg-18/V. 4.0 (see Table 2 for resolution and uncertainties). Balances must be in good operating condition with adequate capacity, sufficiently small resolution, and sufficiently small process standard deviation, as verified by a valid control chart or preliminary repeatability experiments using this procedure.

NOTE 1—Standard deviations obtained from mass calibrations or other weighings often do not reflect the process repeatability of gravimetric calibrations; additional factors such as setting and adjusting the meniscus and process repeatability are normally obtained using this procedure and not those for mass calibrations. The accuracy of the balance and weighing procedures should be evaluated to minimize potential bias in the measurement process.

6.2 Mass standards must be available in nominal values slightly larger than the nominal volume plus mass of the container and calibrated with adequate accuracy. Specification E617 provides specifications and tolerances for mass standards. Mass standards are used as an integral part of this procedure. They may also be used to calibrate or verify the balance prior to use to ensure accurate mass values are used in the calibration. Ordinarily, weights with Class 3 (OIML F1) or better tolerances are required. Uncertainty limits are specified by Specification E617.

6.3 *Thermometer*, for measuring the temperature of the water and air. Thermometers are used for determining the water density, the air density, and the volumetric temperature corrections. The accuracy of the gravimetric volume measurement depends on the accuracy of the temperature standards used (see Table 2 for resolution and uncertainties).

6.4 *Barometer*, capable of providing atmospheric pressure measurements used in determining air density, consistent with the resolution and uncertainty given in Table 2. If barometric pressure is obtained from the local weather service, suitable assurance must be provided to document that the pressures are provided at “station elevations” and not “sea level” corrections; adjustments may be needed for laboratories not at applicable elevations.

6.5 *Timing Device*, for timing delivery and drain times.

6.6 *Distilled or Deionized Water*, suitable for general laboratory purposes, according to Specification D1193 Type III, ISO 3696, Class 3, equivalent or better, measured or calculated to the nearest 0.000001 g/cm³.

TABLE 1 Typical Reference Temperatures for Measured Volumes

Commodity	Reference Temperature ^A
Frozen food labeled by volume (for example, fruit juice)	-18 °C (0 °F)
Beer	3.9 °C (39.0 °F)
Food that must be kept refrigerated (for example, milk)	4.4 °C (40 °F)
Petroleum (International Legal Reference)	15 °C
Distilled spirits or petroleum (U.S. Legal Reference)	60 °F
Compressed Natural Gas (CNG) – International and U.S. Legal References	15 °C (60 °F)
Wine	20 °C (68 °F)
Unrefrigerated liquids (for example, sold unchilled, like soft drinks)	20 °C (68 °F)
Hydrogen (H ₂)	21 °C (70 °F)
Petroleum (Hawaii)	26.67 °C (80 °F)
Recommended for countries by ISO 384 for tropical use	27 °C

^A Values may be legal limits as reference temperatures and not calculated conversion values.

TABLE 2 Measuring Instrument Recommendations

Measurement Parameter and Range	Resolution	Uncertainty ($k = 2$) ^{A,B}
Balance (Nominal capacity > 2000 mL to 10 000 mL)	10 mg (0.01 g)	40 mg (0.04 g)
Balance (Nominal capacity > 1000 mL to 2000 mL)	1 mg	4 mg
Balance (Nominal capacity 10 mL to 1000 mL)	0.1 mg	0.4 mg
Balance (Nominal capacity < 10 mL)	0.01 mg	0.04 mg
Mass standards (See Specification E617 or OIML R111)	ASTM Class 3 OIML Class F1	< 1/3 of specified tolerances
Relative humidity (40 % to 60 %)	1 %	5 %
Air temperature (17 °C to 23 °C)	0.1 °C	0.2 °C
Water temperature (17 °C to 23 °C)	0.01 °C	0.1 °C
Barometric pressure	0.13 hPa (0.1 mmHg)	0.67 hPa (0.5 mmHg)
Timing Device (calibration not required)	1 s	1 s / 24 h

^A Uncertainties for balances are recommended for the maximum capacity of the balance but may be reduced proportionally according to the applicable range of actual use.

^B Uncertainties for other measuring instruments are determined based on the calibration and range of use as required.

6.7 Recommended tolerances and uncertainties for measuring instruments used in this procedure and for the calculation of V_{20} are provided in Table 2.

7. Laboratory Conditions, Cleaning, Setting and Adjusting the Meniscus, Timing Considerations

7.1 This section describes laboratory and instrument conditions and techniques required to obtain accurate and reproducible volumetric measurements.

7.2 Environmental Conditions Needed for Performing Gravimetric Volume Calibrations:

7.2.1 Stable laboratory environments are essential for obtaining accurate measurement results. Balances have manufacturer's normal operating ranges and mass standard and volumetric instruments have applicable reference conditions as well. The following temperature and relative humidity conditions are recommended. Deviations from recommendations require assessment of the impact on measurement results.

7.2.2 *Temperature of the laboratory* should be between 17 °C and 23 °C, stable to ± 1 °C per hour (or during the calibration). The closer measurements are to the reference temperature and standard conditions, errors due to coefficients of cubical expansion uncertainty, and buoyancy errors associated with using balances and mass standards may be minimized. Balances must be within the manufacturer's operating specifications.

7.2.3 *Relative humidity of the laboratory* should be between 40 % and 60 %, stable to ± 5 % per 4 h to meet most balance manufacturer operating conditions. While relative humidity is used and corrected in the calculation of air density, it does not correct for potential errors due to evaporation or condensation. Stable relative humidity may also minimize the impact of evaporation or condensation on the volumetric instruments and can minimize the impact of static electricity on volumetric instruments, mass standards, and balances during the measurements.

7.2.4 *Water and Volumetric Instrument Equilibration*, to laboratory conditions will minimize potential uncertainties in the calibration results due to uncertainties associated with temperature changes during the measurements, water density calculations, and corrections associated with the coefficients of cubical expansion. The length of time required for equilibration is dependent on the starting temperature of the water and

the laboratory conditions. References provide guidance for equilibration times between 2 h and 24 h and depend on the starting temperatures of each item used in the calibration. The recommended difference between laboratory instruments and water is less than 0.5 °C.

7.3 Cleanliness of Volumetric Instruments:

7.3.1 The volume contained in, or delivered by, a volumetric instrument depends on the cleanliness of the internal surface. It is important that volumetric instruments are thoroughly cleaned following standard laboratory procedures or manufacturer's instructions before being calibrated or used. Safe cleaning materials should be used taking care not to use cleaning materials that can damage the instrument. Glass and metal volumetric instruments must be sufficiently clean to permit uniform wetting of the surface. When clean, the walls will be uniformly wetted, and the water will adhere to the surface in a continuous film with no obvious air bubbles. The clean walls of some plastic instruments, however, will not be wetted. Lack of cleanliness causes irregularities in capacity by distorting the water at the surface of the instrument, appearance of air bubbles on the interior surfaces, and can impact the meniscus reading and setting as well as the uniform filling or delivery of water. Regardless of the type of vessel, if chemicals other than water are used for cleaning, always rinse thoroughly, first with tap water and then with distilled water and let dry fully for "to contain" calibrations.

7.3.2 Example cleaning methods are available in NISTIR 7383, GMP 6 and 7, and GLP 13, ISO 4787, Annex A, and EURAMET/cg-21/V. 2.0 (for metal volumetric instruments).

7.4 Reading and Setting a Liquid Meniscus:

7.4.1 *Reading the Meniscus*—For all volumetric instruments calibrated by this procedure, the reading must be made on a level surface or with the instrument level and the reading made on the lowest point of the meniscus. For the lowest point to be observed, it is necessary to place a shade of some dark material immediately below and behind the meniscus, which renders the profile of the meniscus dark and clearly visible against a light background. A convenient device for this purpose is a collar-shaped section of thick black rubber tubing, cut open at one side and of such size as to clasp the tube firmly. Alternatively, black paper may be used. "Short line" graduated vessels where the lines are less than one half of the circumference may be

read more accurately by placing a mirror against the rear of the vessel to reflect the front line.

7.4.2 Setting the Meniscus—Setting of the meniscus shall be performed by one of the two methods detailed below on a level volumetric instrument. Wherever practical, the meniscus should descend to the position of setting.

7.4.2.1 The position of the lowest point of the meniscus with reference to the graduation line is horizontally tangent to the plane of the upper edge of the graduation line. The position of the meniscus is obtained by having the eye in the same plane of the upper edge of the graduation line.

7.4.2.2 The position of the lowest point of the meniscus with reference to the graduation line is such that it is in the plane of the middle of the graduation line. This position of the meniscus is obtained by making the setting in the center of the ellipse formed by the graduation line on the front and the back of the tube as observed by having the eye slightly below the plane of the graduation line. The setting is accurate if, as the eye is raised and the ellipse narrows, the lowest point of the meniscus remains midway between the front and rear portions of the graduation line. By this method, it is possible to observe the approach of the meniscus from either above or below the line to its proper setting.

NOTE 2—The difference between meniscus positions resulting from the alternative methods of adjustment is the volume equivalent of one half the thickness of the graduation line. In the case of volumetric instruments where the capacity is read as the difference between two meniscus readings (for example, on a burette), then no error results if the article is manufactured using one method of adjustment and is later used by the other method. Bias and the impact of visual acuity and lighting when reading the meniscus should be studied by the laboratory to determine differences among operators and may be captured as a component of the calibration uncertainty. Differences in reading the meniscus may be apparent in the results of interlaboratory comparisons, especially when different procedures are selected for use in each laboratory. Calculation of the uncertainty associated with reading and setting the meniscus may be performed using calculations provided in NISTIR 7383, GMP 3, EURAMET/cg-19/V. 3.0, or ISO 4787, Annex B as noted in Section 17.

7.5 Delivery and Drain Times—For volumetric instruments used for delivery of a liquid, the volume delivered is always less than the volume contained, due to the film of liquid left on the inner walls of the volumetric instrument. The volume of this film depends on cleanliness as well as the time taken to deliver the liquid, and the volume delivered decreases with decreasing delivery time. For example, the delivered volume of a pipette or burette will decrease if the tip is broken (shorter delivery time) or will increase if the tip is not clean and the outflow of liquid is restricted. When liquids other than water are used in volumetric instruments, the liquid viscosity will affect delivery times and sample retention on the inside of the volumetric instrument. Delivery times and waiting times are usually specified for volumetric instruments and must be followed.

8. Calibration Procedure for Burettes

8.1 Clamp the burette vertically on a support stand; also clamp a plain glass test tube, large enough to hold a thermometer, near the burette if the burette is of such a size that it is not large enough to insert a thermometer in the top for observing the temperature of the water. Fill the burette from a

reservoir or storage bottle, in which the water has reached equilibrium with room temperature, and check to verify that there is neither leakage from the tip nor from the stopcock plug. Drain and record the delivery time. Delivery time is determined by the unrestricted outflow of the water from the zero mark to the lowest graduation mark with the stopcock fully open. Refill the burette to approximately 10 mm above the zero mark and fill the test tube that holds the thermometer; record the temperature. Set the meniscus on the zero graduation line, using the burette stopcock to lower the liquid level, and touch the tip with the wetted wall of a beaker to remove any excess water. A weighing flask (transfer vessel) that has been tightly stoppered and weighed empty is placed with the inside of the neck in contact with the tip of the burette (the flask will be at a slight angle).

8.2 Fully open the stopcock until the water is only a few millimeters above the graduation being calibrated and then the stream is slowed to make an accurate setting. When the setting has been completed, move the flask horizontally, breaking the contact with the burette. Recheck the setting.

8.3 Then stopper and weigh the flask a second time, after which refill the thermometer tube and calibrate the next interval in the same manner as the first one—from the zero graduation line to the next graduation line needed.

8.4 For burettes with a specified waiting time, use the following procedure: after adjustment to the zero graduation line, fully open the stopcock until the meniscus has reached a position a few millimeters above the graduation line for calibration. After the specified waiting time (for example, 30 s), adjust the meniscus to the graduation line, remove the flask, and weigh.

8.5 Repeat the procedure to obtain replicate values. Report the mean of the calibrated values as the calibrated measurement result. Use the repeatability of the runs to ensure the validity of the measurements according to Section 16 and to determine the uncertainty component associated with the measurement repeatability according to Section 17.

9. Calibration Procedure for Pipettes

9.1 Fill the pipette with distilled water by suction to the higher graduation mark and measure the delivery time with the tip in contact with the glass surface of the internal side of a beaker. Refill by suction slightly above the nominal capacity graduation. Record the temperature of the distilled water in the beaker from which the pipette is filled. Remove any water on the outside of the tip by a downward wipe with filter paper after the filling is completed. Then slowly lower the meniscus to the index using either a stopcock or hose clamp for “fine control.” The tip must be in contact with the wet wall of a beaker while the setting is made on the index line. Do not remove any water remaining on the tip. Hold the pipette in a vertical position and deliver water into a previously weighed weighing flask (transfer vessel) with the tip in contact with the inside wall of the neck of the flask. After the water has ceased to flow, wait 2 s, then remove the pipette from contact with the flask. The flask is now stoppered and weighed with its filled load.

9.2 If the pipette has a lower graduation line, the liquid flow must be nearly stopped a few millimeters above the graduation line. After observing a waiting time, if specified, complete the final setting with the tip in contact with the inside wall of the neck of the flask.

9.3 Repeat the procedure to obtain replicate values. Report the mean of the calibrated values as the calibrated measurement result. Use the repeatability of the runs to ensure the validity of the measurements according to Section 16 and to determine the uncertainty component associated with the measurement repeatability according to Section 17.

10. Calibration Procedure of Flasks (to Contain)

10.1 After cleaning and drying, weigh the empty flask including the stopper. Place an appropriately sized funnel in the flask to discharge the water below the stopper. Fill from beaker or supply line, below the reference line so that the walls of the volumetric instrument considerably above the ring mark are not wetted.

10.1.1 Place the flask under a burette and complete filling and setting the meniscus, taking care not to splash water on the walls, after which place stopper in neck to lessen evaporation and weigh. A pipette or dropper with a finely drawn tip may be used to adjust the meniscus instead of the burette. Determine the temperature by placing a thermometer in the filling beaker or in one which has been filled from the water supply. The temperature may be taken after final weighing by placing a thermometer directly in the flask, provided the flask is of sufficient size to accommodate it. Alternatively, after weighing, the water may be poured into a flask of sufficient size to accommodate the thermometer.

10.1.2 To perform repeated calibrations, two options are allowed:

(1) The flask must be dried between each run. Obtaining an initial dry weight will allow the operator to determine when the flask is sufficiently dry.

(2) Remove a sufficient amount of liquid, measure the temperature of the liquid inside the flask, clean the entire neck so that the walls of the volumetric instrument considerably above the graduation line are not wetted, set the meniscus again taking care not to splash water on the walls and weigh the flask. The initial value of the dried flask is used in all repetitions.

10.2 Repeat the procedure to obtain replicate values. Report the mean of the calibrated values as the calibrated measurement result. Use the repeatability of the runs to ensure the validity of the measurements according to Section 16 and to determine the uncertainty component associated with the measurement repeatability according to Section 17.

11. Calibration Procedure of Flasks (to Deliver)

11.1 Do not dry flasks that are calibrated to deliver prior to the test. Fill the flask to approximately the index line and empty rapidly by gradually inclining the flask to avoid splashing on the walls as much as possible. When the main drainage stream has ceased, the flask will be nearly vertical. Hold in this position for 30 s and touch off the drop of water adhering to the top of the flask. Place a watch glass or plastic cap on the flask

to reduce evaporation and weigh immediately. Take a water temperature reading, fill the flask, and make the meniscus setting on the index line, taking care not to splash water on the walls. Place the same cap or watch glass on the filled flask and weigh. The reverse of this procedure may be used, if desirable.

11.2 Repeat the procedure to obtain replicate values. Report the mean of the calibrated values as the calibrated measurement result. Use the repeatability of the runs to ensure the validity of the measurements according to Section 16 and to determine the uncertainty component associated with the measurement repeatability according to Section 17.

12. Calibration Procedure of Other Volumetric Glassware

12.1 *Measuring Pipettes (and Multi-Graduation Pipettes)*—Measuring pipettes may be calibrated by a similar procedure as described in Section 8, except that the tip must be in contact with the wet wall of a beaker or other vessel when the setting is made on the zero line of a measuring pipette. This includes pipettes that have multiple graduations rather than single graduations (one-mark pipettes).

12.2 *Graduated Cylinders*—Volumetric instruments of this kind may be calibrated by the procedures described in Sections 10 or 11, depending on whether they are identified as to Contain or to Deliver (often designated as TC or TD).

13. Weighing Measurements

13.1 The analytical balance that is used should meet manufacturer's specifications and resolution requirements for the measurement. Balances may be calibrated following Practice E898 (or use EURAMET/cg-18/V. 4.0). Follow the balance manufacturer's instructions and good laboratory practice in making the requisite weighings. Weighings should be made with care and expeditiously to minimize evaporation losses which can constitute a source of error. Care must be taken to gently place the vessel in the center of the balance pan to avoid off-center loading errors. Vessels should not be filled while sitting on the balance pan as that practice can introduce hysteresis errors. The vessels that are weighed should be clean externally and handled carefully to avoid contamination and heat or static transfer. Vessels may be wiped with a clean cotton cloth as required and covered or stoppered whenever possible to minimize evaporation. Handling volumetric instruments and mass standards with clean cotton gloves is a good practice, taking care to prevent wetting or other contamination of the gloves. See NISTIR 6969, GMP 10 to better understand factors affecting weighing operations.

NOTE 3—Balance indications are conventional mass values and based on calibration with known mass standards calibrated to a reference temperature of 20 °C, a reference mass density of 8.0 g/cm³, and reference air density of 0.0012 g/cm³ per OIML D 28. The recorded balance indications provide conventional mass values and are uncorrected for air buoyancy under the conditions of calibration.

13.2 One weighing is required of the mass standard used in this procedure (one-point balance calibration). A mass standard is selected to be slightly larger than the loaded capacity of the volumetric instrument to provide a calibration correction factor between zero and the full mass of the vessel loaded with water

being weighed. First zero the balance and then gently place the mass standard in the center of the balance pan and record the reading as I_M .

NOTE 4—For large volumes, and longer time intervals, mass standards at the loaded and at the empty mass of the volumetric instrument, and buoyancy conditions for filled and empty instruments, may be needed with appropriate modifications in the procedure and associated equations. See NISTIR 7383, SOP 14 for alternative procedures and equations for large volumes. Alternatively, use Practice E898 or EURAMET/cg-18/V. 4.0 and calibrate the balance prior to use and use a factor of 1 in lieu of the value M_S/I_M .

13.3 Two weighings are required of the volumetric instrument or transfer vessel, namely I_L referring to the loaded vessel, and I_E referring to the empty vessel. Normally, I_E and I_L are observed under the same conditions, and are best observed by zeroing the electronic balance prior to each measurement. Both required weighings must be completed in as short a time interval as convenient to ensure that weighings are being made under similar environmental conditions. The air temperature, relative humidity, and barometric pressure should be recorded within or as near to the balance as possible for use in air density calculations. The mass of volumetric instrument or transfer vessel is weighed with both loaded and empty conditions, thus cancelling any corrections associated with the density and buoyancy of the vessel/instrument itself.

14. Temperature and Environmental Measurements

14.1 Measurements of the air temperature, barometric pressure, and relative humidity must be made at nearly the

same time and location as the measurements of the calibration. Measurements immediately before and after the calibration may be used to determine average conditions as well as verification of environmental stability during the calibration.

14.2 Measurements of water temperature must be made to determine the temperature of the water used during the calibration as close to the time of calibration as possible, taking into consideration that the insertion of a thermometer into a volumetric instrument where possible will remove the mass of water being weighed when done prior to the weighing. Care must also be taken to minimize possible gradients in the neck of flasks or volumetric instruments due to handling methods and or due to lack of water equilibration with the laboratory environment. Volumetric instruments will gradually equilibrate with the laboratory environment depending on the mass of water present and can influence measurement results if not made as quickly as possible.

15. Calculations

15.1 Calculate the volume from the weight of the water, contained or delivered, as follows:

$$V_{20} = (I_L - I_E) \left(\frac{M_S}{I_M} \right) \left(1 - \frac{\rho_a}{\rho_S} \right) \left(\frac{1}{\rho_w - \rho_a} \right) [1 - \gamma(t - 20)] \quad (1)$$

15.2 *Water Density Calculations*—Calculate water density using the following equations. Sample calculation results are provided in Table X1.2 for use in verifying calculations.

TABLE 3 Variables and Components Used in Eq 1

Equation Component	Represents
$I_L - I_E$	The balance indication difference, in grams, obtained by subtracting the balance indication in grams associated with the empty weighing volumetric instrument or transfer vessel from that associated with the loaded volumetric instrument or transfer vessel.
M_S	The calibrated mass (true mass) of the mass standards used in the calibration procedure or used in the verification of balance calibration prior to use, obtained or calculated from the calibration certificate.
I_M	The balance indication in grams when a known mass standard is placed on the balance.
$\left(\frac{M_S}{I_M} \right)$	The mass divided by the balance indication produces a “calibration correction factor” for the balance at the maximum point of use. This value becomes 1 when the balance has previously been calibrated and verified prior to use.
$\left(1 - \frac{\rho_a}{\rho_S} \right)$	A density term which requires calculation of air density, ρ_a , and using the known density of the mass standards, ρ_S . The density of mass standards is obtained from the manufacturer or from the calibration certificate; if using a calibrated electronic balance, a value of 8.0 g/cm ³ is used for the density of the mass standards (based on electronic balances being adjusted to indicate conventional mass).
$\left(\frac{1}{\rho_w - \rho_a} \right)$	A density term which requires the calculation of air density, ρ_a , and water density, ρ_w , and corrects for buoyancy differences between the conventional mass value reported at reference conditions of 20 °C and mass density of 8.0 g/cm ³ and the density of water, which is slightly less than 1 g/cm ³ (see Eq 4).
$[1 - \gamma(t - 20)]$	The thermal expansion factor used to convert the volume from the temperature of the measurement in degrees Celsius, to the reference temperature of 20 °C. The symbol γ represents the coefficient of cubical expansion of the volumetric instrument or transfer vessel. Table X1.1 provides a short table of possible coefficient of cubical expansion values. Specific data may be provided by the volumetric instrument manufacturer as well.
$[1 - \gamma(t - t_{ref})]$	The thermal expansion factor used to convert the volume from the temperature of measurement in degrees Celsius, to an alternative reference temperature. The symbol γ represents the coefficient of cubical expansion of the volumetric instrument or transfer vessel.