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Metrological Guidance for the Calibration of Laboratory Glassware

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ABSTRACT

This report provides technical guidance for the calibration of laboratory glassware to help the practitioner achieve traceability to the International System of Units and meet customer quality requirements. The discussion of traceability uses the National Institute of Standards and Technology's seven essential elements of traceability as a framework. The guidance also includes how to determine when calibration is necessary, a discussion of the relationship between tolerances and measurement uncertainty, practical tips, and helpful references. This guidance is intended for United States Department of Energy Contractor Standards Laboratories, but many of the principles and information provided are broadly applicable to other calibration entities and other calibration fields.

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EXECUTIVE SUMMARY

The Primary Standards Laboratory at Sandia National Laboratories is responsible for providing metrology-related guidance to Contractor Standards Laboratories at U.S. Department of Energy Laboratories. This document provides such guidance with respect to calibration and use of laboratory glassware and related equipment for volumetric measurement.

Calibration ensures measurements are metrologically traceable to the International System of Units (SI) which is essential to ensuring quality, managing risk, and meeting customer requirements.

The first question addressed in this report is whether calibration is required. The answer assures that calibration is performed when necessary, and that resources are not wasted performing unnecessary calibration work. The most essential determining factor is whether the volume measurement has the potential to impact the quality of a product or service.

The second question addressed is whether volumetric measurements can be sufficiently accurate to meet the requirements. Measurement uncertainty or tolerances from published standards can be used to estimate achievable accuracy. Gravimetric methods are the preferred alternative for processes requiring accuracy that exceeds the capabilities of volumetric glassware.

If volumetric glassware can meet process requirements and calibration is required, metrological traceability requires seven essential elements (from NIST's GMP 13: Good Measurement Practice for Ensuring Metrological Traceability) that are discussed in the context of glassware calibration:

1. The quantity to be measured or calibrated must be unambiguously defined in terms of SI units, and in a way that can be practically reproduced and measured.
2. There must be an unbroken chain of comparisons going back to accepted national, international, or intrinsic standards, with each link in the chain having all seven essential elements.
3. A documented calibration program must exist that preserves traceability over time by recalibration of standards and equipment.
4. A documented measurement uncertainty must be calculated according to accepted methods and reported so that uncertainty can be calculated for every link in the chain.
5. There must be documented and validated measurement procedures and results.
6. The laboratory performing a calibration for any link in the chain must provide evidence of their technical proficiency.
7. A measurement assurance program must be used to validate the measurement processes and ensure the accuracy of standards used at the time of measurement.

Implementing a process that meets these essential criteria and evaluating the results from upstream links in the traceability chain requires good process understanding and critical thinking.

Other topics addressed in this report include water as a standard reference material for density, cleaning of glassware, communication with customers, calibration certificate requirements, the relationship between tolerances and measurement uncertainty, issues related to radioactive contamination, plastic labware, and the calibration of piston-operated pipettes.

There are many requirements that must be met, but also many options in how they are met. Unless necessary, this report seeks to provide recommendations that allow flexibility. One significant recommendation is to adopt procedures already validated and published by standards organizations rather than developing in-house procedures from scratch.

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ACRONYMS AND TERMS

Acronym/Term	Definition
ASTM	ASTM International (formerly The American Society for Testing and Materials)
CCE	(thermal) coefficient of cubical expansion
CCL	commercial calibration laboratory
CSL	contractor standards laboratory
DI	deionized
DOE	United States Department of Energy
GLP	good laboratory practice
GMP	good measurement practice
GUM	Guide to the expression of uncertainty in measurement
IEC (as in ISO/IEC 17025)	International Electrotechnical Commission
ISO	International Organization for Standardization
JCGM	Joint Committee for Guides in Metrology (provider of the GUM and the VIM)
MPE	maximum permissible error
M&TE	measuring and test equipment
NIST	National Institute of Standards and Technology
NISTIR	National Institute of Standards and Technology Interagency or Internal Report
POVA	piston operated volumetric apparatus
PSL	Primary Standards Laboratory (specifically at Sandia National Laboratories)
QMS	quality management system
SBU	standardize before use
SI	International System of Units
SME	subject matter expert
SOP	standard operating procedure
TC	to contain
TD	to deliver
VIM	International Vocabulary of Metrology

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1. INTRODUCTION

This document provides metrological guidance for the calibration of laboratory glassware for volume, with the goal of metrological traceability. Metrological traceability is a "property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty" [1]. When using the International System of Units (SI), the reference used is common for all users, which enables technology, innovation, quality control, and many other benefits. Metrological traceability enables a user of calibrated glassware to measure a volume and be confident (within the measurement uncertainty) that the volume is what the process designer intended. Metrological traceability is what enables results to be reproducible by independent entities.

The intended audience for this document is Contractor Standards Laboratories (CSLs) at U.S. Department of Energy (DOE) Laboratories. Part of the Primary Standards Laboratory's (PSL's) mission at Sandia National Laboratories is providing metrology guidance to this audience [2]. Many of the principles described, however, are applicable to a broader audience and to calibrating types of measuring and test equipment (M&TE) other than laboratory glassware.

The scope of this document is laboratory volumetric glassware, with some coverage of pipettes, a subset of Piston Operated Volumetric Apparatus (POVA). For other volume measurement equipment, such as that used for legal metrology, the reader is referred to the relevant published standards.

1.1. The Goal of Calibration

Calibration establishes metrological traceability (or simply "traceability") of measurements to the SI. Traceability links measurements to a common reference, and enables many aspects of technology, including interchangeable parts, quantitative communication, and quality control, and allows manufacturers to achieve the designer's intent. Traceability facilitates communication about measurement and the uncertainty of that measurement. For example, a specification can be written by one entity, manufacturing performed by another, and inspection by a third.

Traceability is a component of confirming M&TE is adequate for the task and is essential to ensure quality, manage risk, and meet customer requirements. Therefore, these ends are also the goals of calibration.

1.2. Do You Need to Calibrate?

Calibration done properly is not a trivial undertaking, requiring suitable equipment and facilities, trained personnel, documented procedures, calibrated reference standards, etc. Performing calibration unnecessarily therefore has potential to waste significant resources. On the other hand, calibration when necessary is indispensable for ensuring quality. In 2005, the National Institute of Standards and Technology (NIST) found [3] that a number of laboratories had reported failure rates on new glassware as high as 50 %. Accuracy[1] of volumetric glassware cannot be assumed from manufacturer's specifications or any basis other than calibration.

[1] As used here, "accuracy" could be a tolerance, measurement uncertainty, or some other specification. The term "measurement accuracy" is defined in the International Vocabulary of Metrology (VIM) [1] as "closeness of agreement between a measured quantity value and a true quantity value of a measurand," with a note that "The concept 'measurement accuracy' is not a quantity and is not given a numerical quantity value."

Figure 1 presents a flowchart to determine whether the calibration of laboratory glassware is required to ensure quality. The consideration begins not with the glassware, but by considering the proposed process for which the glassware will be used. If the process does not specify a required accuracy for the volume measurement, then before determining that calibration is not required, consider whether the lack of an accuracy requirement is an oversight. This can be determined by asking whether the volume measurement could affect the quality of a product or service. If the answer is truly no, then calibrating the glassware is not required. If, however, the answer is yes, then the process is missing a volume measurement accuracy requirement. This requirement must be determined before further glassware calibration questions can be addressed. Determining the requirement is outside the scope of this document, but in some cases, it can be derived from the product or service specifications.

If a volume measurement tolerance or uncertainty is specified, calibration is necessary to ensure that requirement is met, but it also must be determined whether glassware can realistically meet this requirement.

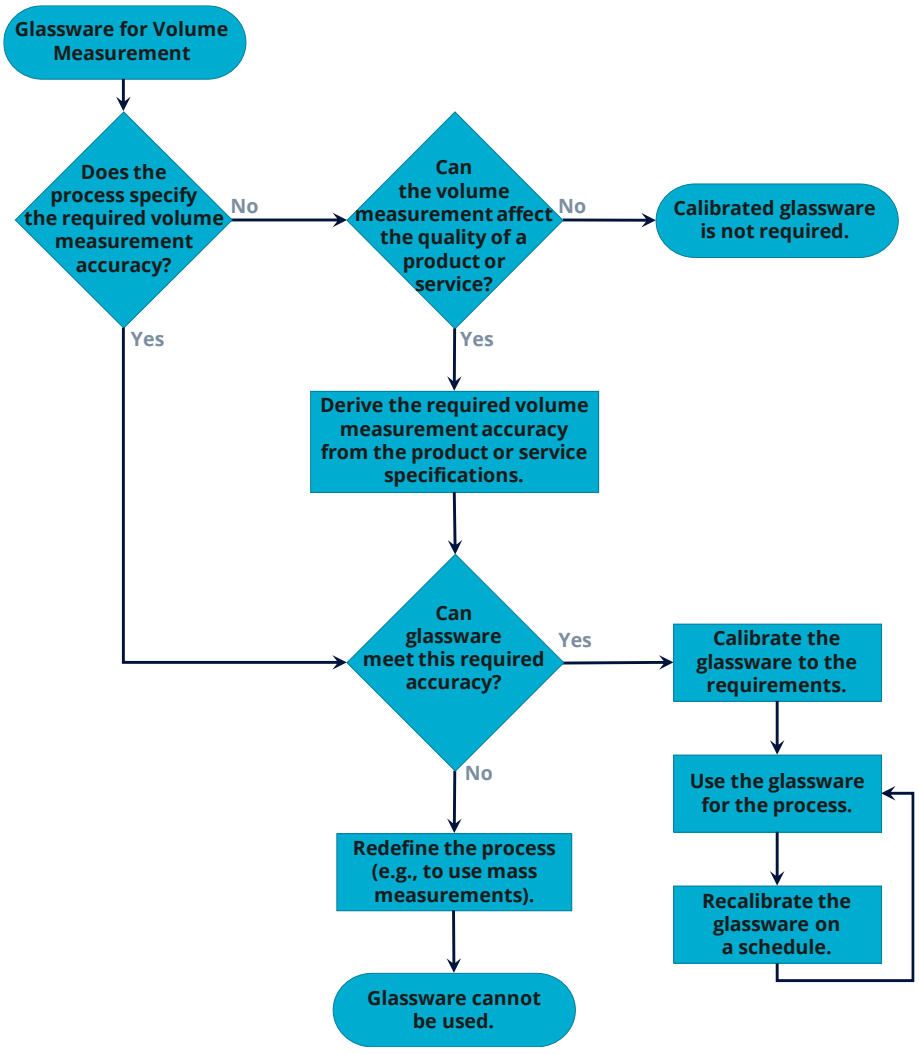

Figure 1. Flowchart to determine whether calibration is required

1.3. Can Volumetric Glassware Meet Process Requirements?

Laboratory volumetric glassware has limitations in achievable measurement accuracy. Factors like reading the meniscus in calibration and use, and measurement uncertainty of the balances and reference weights used in the calibration all contribute to uncertainty of the volume measurement.

As indications of the expected accuracy in normal use, Figure 2 presents Class A glassware tolerances[2] from ASTM standards[3] for selected types of volumetric glassware, ISO[4] maximum permissible systematic errors for piston-operated single channel pipettes, and commercially available[5] gravimetric calibration measurement uncertainties, the latter at an approximately 95 % level of confidence. Note that this information spans several orders of magnitude and is therefore presented on logarithmic axes that visually compress differences between the various data series.

The following observations can be made from Figure 2 (noting that low measurement uncertainty equates to high accuracy):

- The lowest volume measurement uncertainty using glassware is achieved with single-volume items, e.g., volumetric (transfer) pipettes, volumetric flasks.
- The convenience of measuring multiple values of volume with a single glassware item (e.g., a graduated cylinder) comes with a trade-off of higher measurement uncertainty relative to single-volume apparatus.
- Wide-mouth volumetric flasks have higher measurement uncertainty than standard volumetric flasks (for both types as defined in ASTM E288 [4]). The larger diameter at the meniscus location increases sensitivity of the volume measurement to the uncertainty of reading the meniscus. This illustrates that aspects of measurement uncertainty are inherent in the design (or materials) of the glassware. A more accurate calibration cannot lower the measurement uncertainty in use below some threshold related to the design and materials; for example, ASTM Class B glassware cannot be certified to Class A tolerances simply because of a more accurate calibration process (see Section 3.7: Glassware Accuracy Classes).
- Graduated burets have nearly equivalent measurement uncertainty to volumetric flasks with the advantage of graduations but are limited in the range of volumes that can be measured.
- Piston-operated pipettes are the only non-gravimetric option for measuring the smallest volumes. These are typically used over a range from 10 % to 100 % of their maximum capacity, but the ISO 8655 maximum permissible systematic error specification is constant across this operating range [5]. Minimizing the measurement uncertainty requires using the smallest capacity pipette the work allows.
- Gravimetric measurements have the lowest achievable measurement uncertainty for the measurement of a substance's quantity.

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[2] A tolerance may also be called a limit of error, or maximum permissible error (MPE). See Section 3.7.1 for discussion of tolerances.

[3] ASTM International is the current name (since 2001) for the standards organization formerly named The American Society for Testing and Materials (www.astm.org).

[4] ISO is the International Organization for Standardization (www.iso.org).

[5] Examples of measurement uncertainty for commercially available gravimetric calibrations was sourced from the scope of accreditation for one commercial lab [10], and recent calibration certificates reviewed by the PSL (not referenced).

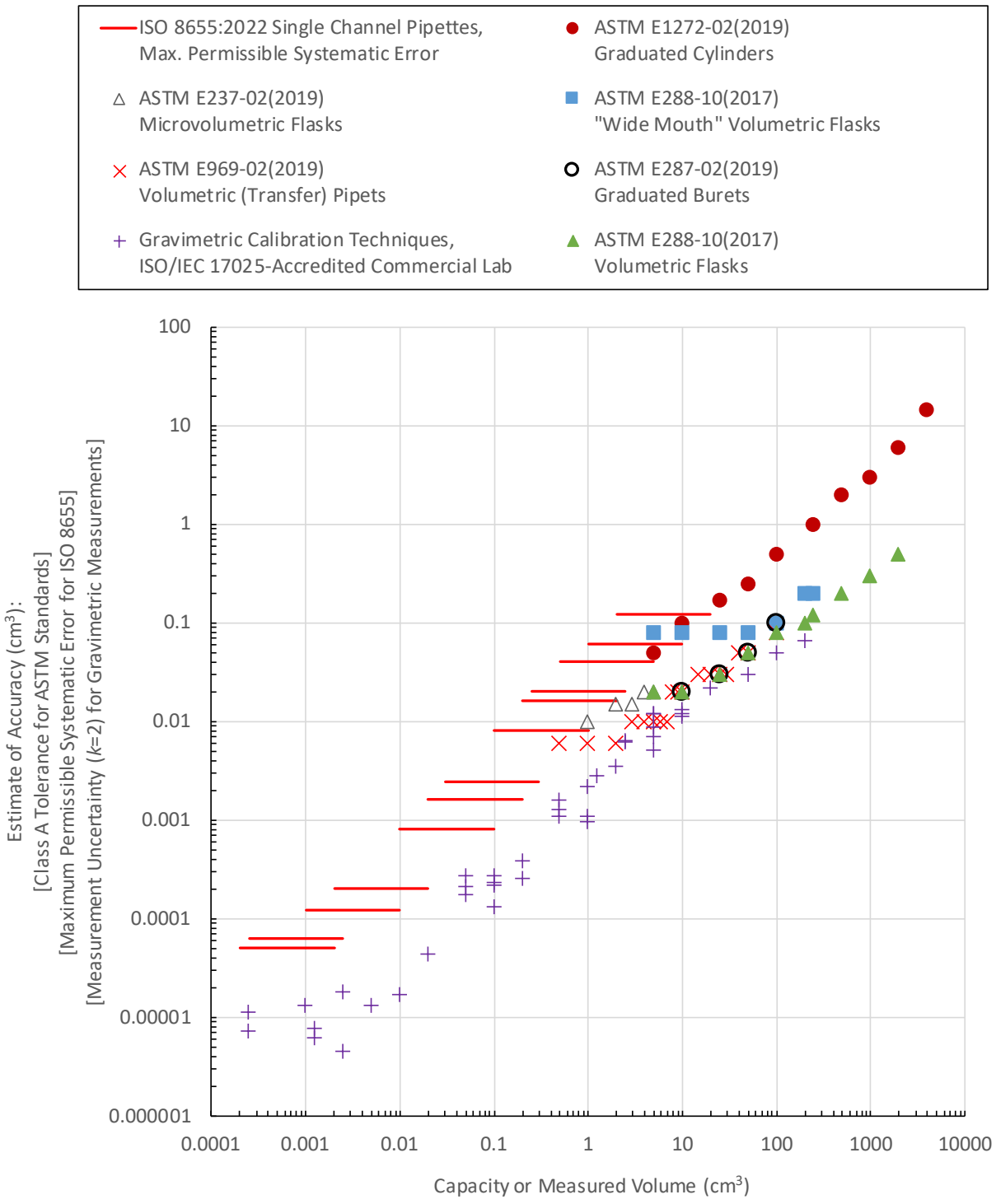

Figure 2. Trends in laboratory volumetric measurement accuracy. Sources: [4], [5], [6], [7], [8], [9], [10], and many individual calibration certificates (unreferenced).

- The measurement uncertainty advantage of gravimetric measurements over volumetric measurements with glassware is relatively greater at smaller volumes.
- Gravimetric measurements can cover the entire range of measured volumes. Data is not shown in the figure beyond 200 cm^3, but for all practical purposes, there is no upper limit.

The volumetric measurement accuracy achievable using laboratory glassware could be estimated using the tolerances published in various standards (such as those referenced in Figure 2). If these tolerances are adequate to meet the requirements, then glassware can be used and shall be calibrated, recalibrated at regular intervals, and calibrated prior to retirement (see Section 2.3: Element Three: Documented Calibration Program).

Alternatively, if the required accuracy is beyond the capability of glassware, then alternative means of measurement are required. Gravimetric measurement is the most likely solution to a need for higher accuracy, and the process should be redefined if necessary to specify this method. The equipment and standards used for the gravimetric measurements shall be calibrated.

If glassware is the selected method, any class of glassware is acceptable if it meets process requirements. ASTM Class B tolerances are generally double Class A tolerances.

Calibration is essential in establishing metrological traceability and falls under the scope of verification[6]. The end user of the glassware should consider the value of additional processes to ensure that the glassware meets the needs of the process (validation[6]).

1.4. The Need for Critical Thinking

There is a large amount of published information on the topics of glassware calibration and metrology from ASTM International, ISO, NIST, and other organizations. This abundance of information does not and cannot remove the need to understand the process and apply critical thinking. This principle is well expressed in the Guide to the expression of uncertainty in measurement (the "GUM"), [emphasis added] [11]:

> Although this Guide provides a framework for assessing uncertainty, it cannot substitute for **critical thinking**, **intellectual honesty** and **professional skill**. The evaluation of uncertainty is neither a routine task nor a purely mathematical one; it depends on detailed knowledge of the nature of the measurand and of the measurement. The quality and utility of the uncertainty quoted for the result of a measurement therefore ultimately depend on the **understanding**, **critical analysis**, and **integrity** of those who contribute to the assignment of its value.

Applied science and engineering involve trade-offs. Gravimetric measurements are more accurate relative to calibrated glassware, but suitable analytical balances may be higher in cost and require more frequent calibration to meet the needs of a given process. Navigating trade-offs requires understanding both the process requirements and characteristics of the options, and applying critical thinking and sound engineering judgement.

There are also potential trade-offs in effort and administrative work for various approaches. Calibration effort can be reduced with longer calibration intervals, but if glassware is found out of

[6] As defined in the International Vocabulary of Metrology [1] verification is "provision of objective evidence that a given item fulfils specified requirements" and validation is "verification, where the specified requirements are adequate for an intended use."

tolerance at a calibration, there is a greater amount of work required to determine the impact on the quality of products and services that are potentially affected.

Determining the required environmental, safety, and health actions is another area that requires process understanding and critical thinking (see Section 3.6).

It is important to be practical. This document seeks to provide insight into the pros and cons with the intent of offering flexibility rather than prescribing a specific option. The goal is to facilitate understanding and critical thinking. Specific budget, resource, or technical constraints may make one option favorable in one circumstance when it would not generally be favorable. For example, plastic labware is generally not desired because of lower accuracy than glassware, but material compatibility may dictate the use of plastic, and this is acceptable provided the impact on measurement uncertainty is appropriately considered.

The published standards may also be written to provide needed flexibility, but in doing so may allow options that increase risk or are not good ideas. For example, ASTM E694 [12] allows for dual-purpose flasks that are "graduated both to contain and to deliver, provided the intention of the different marks is clearly indicated and provided the distance between the two marks is not less than 1 mm." This introduces a risk that if the user is not familiar with the differences between the two markings, the incorrect graduation line may be used.

Sometimes outsourcing of calibration work to commercial calibration laboratories (CCLs) makes the most sense. Generally, CCLs with accreditation to ISO/IEC 17025 [13] can be easily approved as calibration suppliers[7], or nonaccredited CCLs can be audited to confirm compliance in the absence of third-party accreditation. However, no audit can detect every possible issue, conditions can change between audits, and mistakes can happen. Procuring external calibration services is always "buyer beware". It is the responsibility of the calibration lab to provide the supporting evidence of traceability, but it is the responsibility of the customer to assess the validity of the traceability claim (see Good Measurement Practice (GMP) 13 in Reference [14]). Outsourced calibration work and the resulting calibration certificates must be carefully reviewed by a subject matter expert (SME) before acceptance (see Section 3.5.1).

Users and calibrators of glassware should become familiar not only with their own procedures, but also with the relevant published standards. Reading the standards is beneficial for continuing or on-the-job education and to enhance understanding. These documents have been developed by experts in the field, and incorporate lessons learned from decades of experience.

[7] ISO/IEC 17025 accredited laboratories are accredited to a specific scope of measurements. The accredited laboratory may also provide other calibration services outside of this scope. If the approval of a calibration provider is based on the ISO/IEC 17025 accreditation, then only those measurements listed on the scope are approved.

2. SEVEN ESSENTIAL ELEMENTS TO ACHIEVE METROLOGICAL TRACEABILITY

Section 1.1 noted that the goal of calibration is metrological traceability, where measurements are related to references that are common to all users of the SI. NIST published GMP 13 in Reference [14] for ensuring metrological traceability. This document states that "Traceability ensures that the measurements are accurate representations of the specific quantity subject to measurement, within the uncertainty of the measurement." In GMP 13, measurement traceability is characterized by seven essential elements that are discussed below in the context of glassware volume calibration.

Note that many CCLs state their calibrations are "NIST traceable," and yet the calibration certificate lacks evidence of some of the seven elements. If any of the seven elements are missing, traceability is compromised. In addition, "NIST traceable" is incorrect terminology. The correct terminology is "traceable to the SI".

2.1. Element One: Realization of SI Units

This first element of metrological traceability, realization of SI units, requires defining the quantity you seek to measure (called the "measurand"). There are two aspects to this definition: first, that the measurand is defined in terms of SI units so that it is possible to trace the calibration hierarchy back to national, international, or intrinsic standards; and second, that other necessary details of the measurand are defined to ensure there is no ambiguity in the definition.

2.1.1. Defining the Measurand in Terms of SI Units

The SI has seven base units for time, length, mass, electric current, temperature, amount of substance, and luminous intensity, which are respectively the second, meter, kilogram, ampere, kelvin, mole, and candela. Each unit is defined in terms of a fixed value of a physical constant, such as the speed of light in a vacuum. All other SI units are derived by combining products of powers of the base units. For example, the SI unit of force, the newton (symbol N) is defined as a kilogram meter per second squared ($kg \cdot m \cdot s^{-2}$).

The United States is officially a "metric" country, meaning the SI is the unit system in use. All non-SI units (such as pounds for mass and inches for length) are now defined in terms of conversions from SI units. NIST has published a guide to using the SI [15] that includes the values of these conversion factors.

The SI unit of volume is cubic meter (m^3), and by using the standard SI prefixes, various submultiples of this unit are used to suit the application, such as the cubic decimeter (dm^3) or cubic centimeter (cm^3). The unit of "liter" (or "litre" outside the United States) was redefined in 1964 by The Twelfth General (International) Conference on Weights and Measures as a "special name for the cubic decimeter" and permitted its use except in association with measurements of the highest precision [16]. The calibration of volumetric glassware is not one of these applications, so for calibrating glassware, the units of liter (L) and milliliter (mL) can be used in place of cubic decimeter and cubic centimeter, respectively, and any difference between pre-1964 and post-1964 liters is negligible[8]. Glassware may be marked with either mL or cm^3 [16].

[8] NIST has provided guidance [15] that in the United States, the symbol for the liter is uppercase (L, not l) to avoid confusion with the number 1 (one).

Table 1 shows how the units compare at various orders of magnitude and illustrates why, for example, milliliters are most practical for many laboratory glassware items and microliters are commonly used for piston-operated pipettes.

Table 1. Equivalent volumes in different SI units

Cubic Meters (m^3)	Liters (L) Cubic Decimeters (dm^3)	Milliliters (mL) Cubic Centimeters (cm^3)	Microliters (µL)
1	1 000	1 000 000	1 000 000 000
0.001	1	1 000	1 000 000
0.000 001	0.001	1	1 000
0.000 000 001	0.000 001	0.001	1

Traceability of glassware volume depends primarily on mass measurement (in kilograms [kg] or grams [g]), the density of pure, air-saturated water (in kg/m^3 or g/cm^3) as given by an accepted temperature-dependent calculation, and temperature of the water (usually measured in °C rather than K). Mass traceability goes back to national mass standards, and temperature traceability goes back to intrinsic standards, such as fixed-point temperature cells. Information on the accepted equation for density of air-saturated water as a standard reference material is given in Section 3.1.

2.1.2. Defining the Measurand to Remove Ambiguity or Provide Clarity

There are multiple factors that can affect the volume of laboratory glassware, such as temperature (because the glassware expands with temperature, changing the volume), the method by which the meniscus is read, and others. If these factors are not included in the definition of the measurand, the definition becomes ambiguous. Two important principles in calibration are:

1. Items should be calibrated in a manner that is representative of their use.
2. Factors that affect the calibrated value (or measurand) should be measured, controlled if possible, and values reported with the calibration data so the effect of any differences between calibration and use conditions can be quantified and, if appropriate, corrected.

Principle 1 necessitates good communication with the customer when defining the calibration requirements, and Principle 2 necessitates a calibration certificate that thoroughly documents the calibration (see Section 3.5.1). Because of these two principles, the measurand definition for glassware volume includes at least the following items:

- The temperature at which calibration is performed. 20 °C is the reference temperature for laboratory glassware. Other applications (e.g., liquid fuels, beverages, etc.) may have different reference temperature: see References [17] and [18] for details.
- The method by which the meniscus is read (see Section 2.5).
- Whether the glassware is "to contain" (TC) or "to deliver" (TD)[9].
- The delivery time and drain time if the glassware is "to deliver". For example, for volumetric flasks calibrated "to deliver" according to ASTM E542 [18], the delivery time is "rapidly", and the post-delivery drain time is 30 seconds.
- The liquid being used (for laboratory glassware, this will be pure air-saturated water).

[9] The international equivalents of TC and TD are IN and EX, respectively [5].

2.2. Element Two: Unbroken Chain of Comparisons

With a measurand defined so it can be traced back to accepted national, international, or intrinsic standards, the second element requires that it is traced back to these standards through a documented and unbroken chain of comparisons (or calibrations). This is often referred to as a traceability chain or hierarchy, and each link in the chain is required to have all seven essential elements of traceability. In reality, the chain is a network of paths rather than a single chain, but all paths in the network need to be traceable back to the accepted standards.

It is usually not practical for an individual user to trace the chain back to the source, so in practice the user usually evaluates only the previous one, or perhaps two, links in the chain, most often by ensuring the calibration provider conforms to the requirements of ISO/IEC 17025 (see Section A.3 of Reference [13]).

With respect to measurement uncertainty, an important characteristic of a traceability chain is that each link in the chain contributes to the measurement uncertainty of dependent links in the chain. To minimize measurement uncertainty traceability, chains should be kept as short as practical. A principal reason why gravimetric methods have lower uncertainty than volumetric methods is that the volumetric methods are calibrated by gravimetric means, and therefore directly using gravimetric methods eliminates a link in the traceability chain with the associated uncertainty addition.

2.3. Element Three: Documented Calibration Program

Calibrated items need to be recalibrated at appropriate intervals to preserve their metrological traceability over time and use. It is possible for glassware to gradually change dimensionally over time, or because of the use conditions. A documented calibration program defines the calibration intervals and, if appropriate, criteria for reinspection, recalibration, or retirement. Documenting calibration intervals (or due dates of the next calibration) is a requirement of ISO/IEC 17025 [13].

In the past, guidance provided by the PSL[10] suggested that under some conditions, ASTM Class A glassware could be used without calibration, and that certain glassware did not require recalibration. *This guidance is no longer accepted.* Instead, the need for calibration should be determined as described in Section 1.2, and any glassware identified as requiring calibration will also require periodic recalibration and a final calibration before retirement. This latter "closeout calibration" determines whether the glassware volume remained within its certified tolerance throughout its use in the last calibration interval. Any glassware already in use and not being calibrated based on outdated guidance should be reevaluated to determine whether current guidance requires calibration.

Calibration intervals should be determined with consideration for the conditions and frequency of use, in addition to the consequences if the glassware is found out of tolerance. Harsh conditions of use and high frequency of use correspond to shorter calibration intervals. If out-of-tolerance glassware would have severe consequences, more frequent calibration enables earlier detection and the ability to recall or rework products before a negative impact is realized by the customer. Early recalibration should always remain an option; for example, at the completion of a critical project, it may be prudent to recalibrate immediately rather than wait for the end of the calibration interval.

Early recalibration is also appropriate whenever inspection before use identifies any chips, cracks, etching, frosting, or any other obvious damage or defect. In legal metrology [16], inspection before use is mandatory. In the analytical laboratory, inspection before use is strongly recommended. Comparisons with other standards or check standards (see Section 2.7) may help determine which

[10] Memorandum dated August 9, 1995: *Metrology Guidance for the Calibration of Laboratory Glassware*

glassware needs early recalibration and can also reduce the scope of an impact analysis and potential for recalled work should glassware break preventing proper closeout calibration.

The longest calibration interval should be no longer than 5 years [2]. In the 2021 revision of NIST Handbook 105-2 [16], the maximum recommended interval was changed from 10 years to 5 years "for consistency with ASTM and ISO standards". Justifying general use of longer intervals than 5 years (as opposed to interval extensions, discussed below) is often motivated by a desire to avoid calibration. Such justification requires data that demonstrates stability over time, and even if stability is demonstrated still involves an extrapolation into the future, with associated risk. The best way to obtain the data needed for such a justification is calibration, the very activity the justification seeks to avoid. Coleman and Harris note that it is "extremely difficult to justify calibration intervals longer than ten years" [3]. It is therefore recommended that users do not seek to avoid calibration when the criteria in Section 1.2 indicate it is needed. Calibration lowers risk and has accepted methods, whereas justification of longer intervals may have difficulty standing up to peer-review.

Regular calibration has the benefit of generating data that allows monitoring of out-of-tolerance rates to achieve reliability targets. The DOE targets 95 % reliability rate at the end of a calibration interval. Deviation from the reliability target signals a need to adjust assigned uncertainties (or tolerances) or calibration intervals accordingly.

Extending the calibration interval of individual items to suit a particular set of circumstances (such as the need to complete a particular project in progress) involves some risk, but less than the general or widespread use of longer calibration intervals. Extensions are permissible when needed, but should require justification, such as evidence and analysis of stability [18]. Extensions should be no longer than needed and shall comply with relevant organization-specific policies.

Severe use conditions for glassware that indicate shorter intervals or early recalibration are appropriate include [17] [18]:

- Exposure to hydrofluoric acid (HF)[11]
- Exposure to hot phosphoric acid
- Exposure to strong or hot alkalis
- Heating above 150 °C when dry

In general, the ultimate responsibility for determining calibration intervals rests with the owner/user of the calibrated items, who is also the most familiar with the conditions of use. ISO/IEC 17025 requires in Section 7.8.4.3 that "A calibration certificate or calibration label shall not contain any recommendation on the calibration interval, except where this has been agreed with the customer" [13]; however, in practice this requirement is seldom followed by CCLs. DOE policy states the responsibility to set calibration intervals rests with the organization performing the calibration (or outsourcing the calibration to an external lab) [2]. This requires communication with the customer to learn the conditions of use prior to selecting an appropriate interval.

2.4. Element Four: Documented Measurement Uncertainty

Measurement uncertainty characterizes a range in which the true value of a measurand is believed to lie. All calibration results in a traceability chain must be documented, with an associated

[11] Glassware has been observed to change with exposure to HF without visible etching. Washing in base baths has also been observed to change glassware size, but usually with visible etching. (Communication with Dave Wheeler, Sandia National Laboratories, 2021)

measurement uncertainty. Additionally, measurement uncertainty is always stated with a specific level of confidence (commonly ~ 95 %, which for a normal probability distribution corresponds to a coverage factor of k=2). For example, a calibration measurement of a nominally 1 L glass volumetric flask may be reported as "999.98 mL with an expanded uncertainty (k=2) of 0.10 mL, which represents a level of confidence of approximately 95 %". This means the calibrating laboratory is 95 % confident that the volume measured at the time of calibration lies in the 999.88 mL to 1 000.08 mL range.

Measurement uncertainty combines the result of several contributing factors, which includes the following for calibrating glassware, listed in descending order of their usual importance [18]:

- The accepted standard deviation of the calibration process[12]
- Uncertainty due to operator effects (e.g., reading of the meniscus[13,14])
- Uncertainty of water temperature measurement
- Uncertainty of water density
- Uncertainty due to thermal expansion of the glassware[15]
- Uncertainty of the standard masses or calibrated balance[16]
- Uncertainty in the density of standard masses
- Uncertainty in the air density due to measurement of air temperature, pressure, and relative humidity
- Uncertainty due to drain time effects
- Uncertainty due to evaporation[17]

Some factors contributing to uncertainty, such as the uncertainty of the standard masses, depends on the uncertainty of the previous link in the traceability chain. Hence, every metrologically traceable measurement requires knowledge of the uncertainty from its traceability chain. In addition, every measurement has its own contributing factors (e.g., repeatability or standard deviation of the calibration process). The net result is that uncertainty increases along a traceability chain the further the measurement is from the standards at the beginning of the chain.

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[12] The standard deviation used in the uncertainty analysis must be specific to the process. A volume calibration that involves reading a meniscus will have a higher standard deviation than a mass calibration using the same balance. See ASTM E542-22 6.1 Note 1 [18].

[13] The annex of ASTM E694 [12] details the relationship between volumetric error and diameter at the meniscus. This reference is useful when determining uncertainty and tolerances for custom glassware where suitable tolerances are not given by any published standard. Additional useful references for this topic are listed in ASTM E542 Section 16.2 [18].

[14] Individual staff members can be very repeatable reading a meniscus, but there may be systematic differences between staff members. Uncertainty estimates should account for both variability in individual staff members' results and variability between staff members. See ASTM E542 7.4.2.2 Note 2 [18].

[15] ASTM E542-22 Table 6 [18] suggests a range for the thermal coefficient of cubical expansion (CCE) uncertainty of 5 % to 10 % of the value.

[16] The uncertainty of a balance is typically larger than its resolution (see example values in Table 2 of Reference [18]).

[17] Evaporation is a more important consideration for smaller volumes (with a larger surface area to volume ratio) and may be negligible for many volumes. This contribution is determined experimentally and can be minimized with procedural steps, such as covering the container during weighing. ASTM E542 suggests correction for evaporation becomes important for volumes below 0.1 cm^3 [18]; however, this standard does not provide the necessary methods and corrections.

If any link in the chain is missing documented quantification of the measurement uncertainty, all dependent links will lack metrological traceability.

The analysis and expression of measurement uncertainty shall be done in accordance with internationally accepted methods given in Reference [11]. There is no need to start an uncertainty budget from scratch, as References [18] and [17] give example uncertainty budgets for calibrating glassware. These examples are in accordance with the accepted methods. Critical thinking with understanding of the process still needs to be applied to ensure that the contribution of various factors is not duplicated (see Note 5 in Reference [18]).

Note that tolerances are not the same as measurement uncertainty. For a discussion of tolerances and their relationship to uncertainty, refer to Sections 3.7.1 and 3.7.2.

2.5. Element Five: Documented Measurement Procedure

The fifth essential element of metrological traceability is that the calibrations performed at each step in the traceability chain must have used documented and validated procedures, with the measurement results and associated uncertainty documented on a calibration certificate.

Not all procedures have equivalent performance due to differences such as the number of measurement repetitions or ability to cancel drift in the process. Validating a procedure not only includes ensuring that the measurement uncertainty achieved is sufficient to meet customer needs, but also that the procedure produces the correct result. Validating a new procedure can be a substantial effort requiring, for example, a new uncertainty analysis supported by experimental data, and interlaboratory comparisons. Given the effort required to properly institute a new procedure, using an existing accepted or published and validated procedure whenever possible is strongly recommended. Taking this approach saves resources and facilitates interlaboratory comparisons with other labs using the same method. Whenever a published standard is used, confirm that the version in use is the current active version.

Standard Operating Procedure (SOP) 14 in NISTIR 7383 [17] and ASTM E542 [18] are accepted and validated practices or procedures and are recommended for the gravimetric calibration of laboratory glassware. In function, they are equivalent; however, the NIST documentation tends to have more detailed procedural steps and practical advice. Calibration labs would benefit from familiarity with both documents.

Besides SOP 14, NISTIR 7383 [17] and NISTIR 6969 [14] contain other necessary SOPs, Good Laboratory Practices (GLPs), and GMPs that are essential for a complete volume calibration capability.:

- In NISTIR 7383:
  - GLP 10 Good Laboratory Practice for the Purity of Water (includes expressions to calculate air-saturated water density as a function of temperature)
  - GLP 13 Good Laboratory Practice for Drying "To Contain" Volume Standards
  - GMP 3 Good Measurement Practice for Method of Reading a Meniscus Using Water or other Wetting Liquid
  - GMP 7 Good Measurement Practice for Cleaning Precision Glassware
  - SOP 14 Recommended Standard Operating Procedure for Gravimetric Calibration of Volumetric Standards Using an Electronic Balance
  - SOP 17 Standard Operating Procedure for Control Charts of Check Standards

    - SOP 20 Standard Operating Procedure for Standard Deviation and Range Charts
- In NISTIR 6969:
    - GLP 1 Good Laboratory Practice for the Quality Assurance of Laboratory Measurement Results
    - GMP 10 Good Measurement Practice for Understanding Factors Affecting Weighing Operations
    - SOP 1 Recommended Standard Operating Procedure for Calibration Certificate Preparation
    - SOP 2 Recommended Standard Operating Procedure for Applying Air Buoyancy Corrections
    - SOP 4 Recommended Standard Operating Procedure for Weighing by Double Substitution
    - SOP 9 Recommended Standard Operating Procedure for Control Charts for Calibration of Mass Standards
    - SOP 29 Recommended Standard Operating Procedure for the Assignment of Uncertainty
    - SOP 30 Recommended Standard Operating Procedure for a Process Measurement Assurance Program

Examples of information required in procedures that is available in the NIST documents above include:

- Environmental control requirements
- Equipment and standards with associated measurement uncertainty requirements
- Inspection criteria for items being calibrated
- Procedural steps
- Calculation or analysis documentation
- Measurement assurance instructions and documentation (see Section 2.7)
- Measurement uncertainty analysis documentation

Some information not contained in the NIST documents are tolerances for various types of glassware. This information is found in the relevant standards specific to the apparatus type such as the ASTM and ISO standards referenced in Figure 2. The International Organization of Legal Metrology (OIML)[18] is another organization with relevant published standards.

While the documents listed above provide almost all the procedure documentation needed for volumetric glassware calibration, a calibration laboratory that adopts these procedures will still need their own internal procedure with site-specific requirements such as the specific standards to be used, the number of measurements to be made, record keeping procedures, safety requirements, etc. Where the above procedures have more than one option, the site-specific procedure shall specify which option is to be used, or if more than one may be used, the circumstances under which each is used.

[18] www.oiml.org

An example is the method of reading the meniscus. Both GMP 3 and ASTM E542 have two options for reading the meniscus. The choice of method may depend on how far around the vessel perimeter the graduation lines extend and whether the liquid is opaque or transparent. Most importantly, the method used in calibration should be consistent with how the glassware is used. Communication with the customer to review meniscus reading methods is key to a quality calibration result. Different customers or items may require different methods, so the calibration staff must be proficient in both methods.

Another example is using either a calibrated balance or mass standards as the reference. ASTM E542 Section 17.7 notes that when using a calibrated balance under conditions that differ "significantly" from the reference conditions for conventional mass (i.e., 20 °C, 8 g/cm^3 reference density, 0.0012 g/cm^3 reference air), additional uncertainty evaluation should be considered [18]. "Significantly" is not quantified, so to avoid the additional evaluation, if there is a question as to whether the conditions are sufficiently close to conventional mass reference conditions, mass standards should be used as the reference rather than a calibrated balance. This recommendation applies to laboratories located at high altitude, for example.

NISTIR 7383 contains a procedure for calibrating glassware by a volume transfer method; however, it is simpler for a glassware calibration lab to calibrate all glassware gravimetrically. The benefits include having fewer procedures and calibration standards to maintain, and a shorter traceability chain with associated lower measurement uncertainty. It does require staff that have training specific to gravimetric calibrations [3]. Examples of such specific training are determining an effective density (not average density) when summations of weights are used and understanding that the NIST and ASTM procedures use true mass (mass in a vacuum) in the analysis, not conventional mass.

Users of glassware should also have some familiarity with the calibration procedures. ASTM E542 contains many best practices for use of volumetric glassware, for example, pipetting onto wetted walls; using specified and consistent wait times when emptying; avoiding liquid splashes above the graduation line; using a burette stopcock fully open until close to the target volume target, then slowing the flow; etc.

Procedures are a foundational piece of a calibration capability. Control charts (Section 2.7) cannot be populated with data without an established procedure. Uncertainty analysis (Section 2.4) cannot be completed without the process standard deviation from the control charts. All of these are prerequisites to the next element: Accredited Technical Competence.

2.6. Element Six: Accredited Technical Competence

Just as metrological traceability requires documented measurement uncertainty for all steps in the chain, the laboratories performing the calibrations at each step of the chain must be technically competent and have evidence of the same. Suitable evidence includes appropriate training records, results of proficiency tests or interlaboratory comparisons, and accreditation to ISO/IEC 17025 (the international standard for competence of testing and calibration laboratories) from a recognized accreditation body. Generally, this means an accreditation body that is an ILAC[19] signatory.

As mentioned in Section 1.4, it is the responsibility of the calibration laboratory to provide the evidence of traceability (and technical competence), but it is the customer's responsibility to assess

[19] ILAC is the International Laboratory Accreditation Cooperation. Signatories to the ILAC Mutual Recognition Arrangement (MRA) can be found at https://ilac.org/

this evidence and the validity of the traceability claim. It is not unknown for an accredited laboratory to provide calibration services that fall short of ISO/IEC 17025 requirements, so no calibration certificate should ever be blindly accepted without thorough review.

2.7. Element Seven: Measurement Assurance

The final element of metrological traceability is a measurement assurance program[20] that ensures (1) the validity of the measurement process and (2) the accuracy of reference standard(s) used at the time the measurements are performed.

Various tools can be used to form a measurement assurance program, such as:

- Repeated measurements, which enable monitoring the statistical control of a measurement process using standard deviation charts (preferred) or range charts. (See SOP 20 in Reference [17].)
- Control charts of check standard measurements, where the check standard is representative of the items being calibrated and is measured by the same process. (See SOP 17 in Reference [17].)
- Software quality assurance to ensure that calculations performed by software are the correct calculations for the intended purpose (validation) and are performed correctly (verification).

For the first two items, note that SOP 14 [17] calculates the average volume from two runs, but the procedure notes that NIST calibrations generally use five replicates. A different number of replicates is a different process, and a standard deviation chart, range chart, and control chart can only be used for data coming from the same process. For all data on these charts, the number of replicates must be fixed by the procedure.

A control chart of check standard measurements can be interpreted to provide assurance that significant changes in systematic error or process variability have not occurred (an internal check). In addition, check standards used for control charts should ideally have an independently measured reference value (i.e., a measurement value from an independent calibration laboratory) that can be compared to the mean value of the control chart data to ensure agreement within measurement uncertainty (an external check).

The calibration laboratory should have an established process for software quality assurance. This process could include comparisons of calculation results with results of independent calculations. The appendix of ASTM E542 [18] includes example values from the water and air density calculations that could be used for this purpose, as does GLP 10 [17] (water density only).

[20] Despite qualitative similarity in the names, a measurement assurance program is not the same as a quality management system (QMS). A measurement assurance program is specific to the calibration process(es) whereas a QMS is a formal system that documents responsibilities, processes, etc. to achieve quality objectives for the laboratory. The QMS has wider scope that encompasses other business functions such as shipping and receiving, administration, etc.

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3. OTHER CONSIDERATIONS

3.1. Water as a Standard Reference Material

The volume of calibrated glassware is calculated as mass divided by density of the water weighed in the process. The water serves as a standard reference material, and therefore the purity or quality of the water is important in gravimetric calibrations (pure water is not required for volume transfer calibrations, but gravimetric calibrations are recommended over volume transfer methods – see Section 2.5).

There are multiple options to achieve the required quality, and selecting a suitable system depends on the purity of the source water. Distillation, reverse osmosis, ion exchange, or combinations of these processes, followed by filtration, are suitable methods. Tap water and filtration alone are, however, inadequate, because the water purity requirements are such that both solid and chemical contaminants must be removed. GLP 10 in Reference [17] has substantial practical advice for the topic of water purification, and states that while not directly correlated to density, electrical conductivity of ≤ 5 μS/cm (electrical resistivity of ≥ 0.2 MΩ·cm) is recommended as adequate for gravimetric glassware calibrations. ASTM E542 [18] requires water that meets ASTM D1193 Type III or ISO 3696 Class 3 specifications (or better).

Water systems should be properly maintained, and checks should be performed to confirm the water quality. Checks could include conductivity or resistivity measurements, or use of a "quality assurance reference flask" (direct measurement of density) (GLP 10 in [17]).

Another alternative to controlling water quality is measuring the density of an uncontrolled water source using a calibrated 5 or 6 place density meter. However, this equipment is likely to be more costly than controlling the water quality and will itself require calibration [3]. This approach may also cause contamination of the customer's glassware[21] and is therefore not recommended.

Water temperature accuracy is as important as water purity for a correct density determination. SOP 14 states the requirements for the water thermometer as "resolution and uncertainty less than 0.1 °C", and ASTM E542 requires resolution of 0.01°C and uncertainty (k=2) of 0.1 °C. If water is coming straight from a tap through the purification system and into the calibration process, the water temperature may fluctuate significantly more than the desired uncertainty. Storing enough purified water in the lab to complete a calibration is therefore critical to the quality of the process. This stored volume of water should be allowed sufficient time to equilibrate with the lab environment prior to use[22].

After equilibration, it is assumed that the water is air-saturated. The currently accepted expressions to calculate the density of air-saturated water are originally sourced from Tanaka et al. [19] and are duplicated in both ASTM E542 [18] and SOP 14 [17]. As mentioned in Section 2.7, the software (e.g., a spreadsheet) used to calculate water density should be validated using example calculation results that are available in the latter two references.

3.2. Cleaning Glassware

It is the responsibility of the customer to deliver glassware to the calibration lab free of contamination (biological, chemical, radiological, and other contamination). It is the responsibility of

[21] For customers with glassware used in semiconductor fabrication processes, deionized (DI) water is required in calibration to prevent contamination.
[22] ASTM E542 has a recommended difference between laboratory equipment and water of less than 0.5 °C [18].

the calibration lab to clean the glassware for the calibration process, as described below. The calibration lab and customer should communicate to ensure that the decontamination and cleaning processes in each lab do not affect the operations of the other. For example, detergents containing phosphates can leave a hydrophobic deposit on the glass that is hard to remove and can make setting the meniscus difficult (a problem for calibration and use). Some candidate cleaning processes may leave deposits that affect chemical analysis operations. Good Measurement Practice for Cleaning Precision Glassware (GMP 7 in [17]) has plentiful information for this topic and is recommended reading. Consult a chemistry SME with specific questions not addressed in this reference.

Glassware to be calibrated should be cleaned and dried one to two days in advance of the calibration to allow the item to equilibrate with the lab environment. Final rinsing at the end of the cleaning process must be with pure distilled or deionized water (i.e., the same water source used for calibration) (GMP 7 in [17]). After cleaning and drying, the vessel should be covered to minimize contaminants (e.g., dust) collecting inside. General good housekeeping in the lab is always recommended and will help minimize contamination after cleaning and drying. After equilibration, it is recommended to obtain an initial baseline dry weight to use as a reference, for example after any time the vessel is dried in the calibration process (GLP 13 in [17]).

Cleanliness of both the inside and outside of the vessel is important as any removal of dust or other material during the calibration could cause measurement error (GLP 13 in [17]). For the same reason, the outside of the vessel must be dry during all weighing operations.

Bubbles, foam, and splashes onto the walls above the graduation line can all cause measurement errors. Proper cleaning and liquid transfer techniques help prevent these issues.

Glassware should never be dried by heating above 150 °C (see Section 2.3). Autoclaves (steam sterilizers) are acceptable to use with calibrated glassware (GMP 7 in [17]).

3.3. Laboratory Facilities and Layout

Liquid temperature and balance stability are critical factors in gravimetric calibration, and therefore the laboratory heating, ventilation, and air conditioning (HVAC) system plays a critical role in the quality of calibrations. Environmental requirements differ only slightly between ASTM E542 and SOP 14, and are given in Table 2. Gradients or changes in environmental conditions can be minimized by minimizing the total process time within the constraints of the procedure.

Table 2. Required laboratory environmental conditions

Parameter	ASTM E542 Requirements	SOP 14 Requirements
Laboratory Temperature	17 °C to 23 °C Stable to ± 1 °C/h (or during the calibration)	18 °C to 23 °C Stable to ± 1 °C/h, during the calibration
Relative Humidity	40 % to 60 % Stable to ± 5 % / 4 h	40 % to 60 % Stable to ± 10 % / 4 h

The layout of the calibration work area needs careful consideration. Water should be stored near to the measurement area to minimize differences due to spatial temperature gradients, but with sufficient spacing to avoid spilling water on the balance during liquid transfer steps. Calibration work can be repetitive, so the ergonomics of the work area are important to minimize fatigue and risk of worker injury.

Tools for reading the meniscus should be readily available (suitable lighting, meniscus readers (see GMP 3 in [17]). While only some calibration personnel may require additional tools, such as magnifiers, such tools should be made available to all calibrators, as they make the process easier for all and will likely improve quality.

3.4. Communication with the Customer

Communication with the customer is given its own section in this document because it is so critical to successful calibration. Communication is mentioned in this document in the following contexts:

- Section 2.1.2: Defining the customer's calibration requirements
- Section 2.3: Setting calibration intervals with input from the customer on conditions of use
- Section 2.5: Coordinating the meniscus reading method used to ensure consistency between calibration and use
- Section 3.2: Coordinating decontamination (to ensure safety) and cleaning methods to ensure process compatibility with both calibration and use
- Section 3.5: Completing documentation on the calibration certificate to ensure that the customer has all the information required for metrological traceability
- Section 3.7.2: Obtaining agreement with the customer on the decision rule and communicating all information necessary for them to assess risk

Communication can be facilitated by providing a form for the customer to complete when submitting glassware for calibration. Such a form allows the customer to specify their calibration requirements (including the decision rule for compliance with tolerances), certify that the glassware is free from contamination, and provide other information.

3.5. Documentation

Documentation is critical to calibration and metrological traceability: all seven essential elements of metrological traceability have a documentation component. Any time a decision or assumption is made it should be documented. Suitable documentation avoids rework, confusion, and ambiguity, and facilitates transparent communication. Two calibration-specific types of documentation are discussed in this section: calibration certificates and equipment (glassware) markings.

3.5.1. Certificate Requirements

It is the responsibility of the calibration laboratory to provide all supporting evidence of metrological traceability [14]. To fulfill this responsibility and minimize the possibility of misunderstanding or misuse, the calibration certificate shall include all information required by ISO/IEC 17025 Section 7.8 [13]. SOP 1 in Reference [14] includes an appendix with a checklist of these items[23]. In addition to the ISO/IEC 17025 requirements, information specific to glassware volumetric calibration shall also be included on the certificate if applicable (most of these are listed in SOP 14 in [17]):

- Whether the volume is "To Contain" or "To Deliver".
- Delivery and waiting/drain times if the volume is "To Deliver".

[23] This checklist is a highly recommended tool both for design and preparation of calibration certificates and to facilitate and document reviews of certificates from CCLs.

- The method by which the meniscus was read (the name of the method is sufficient).
- The reference temperature.
- The standard reference material (or liquid) used.
- The thermal coefficient of expansion and whether this value was assumed or measured. A value should be provided in the manufacturer's documentation.
- Construction (i.e., material and design of the glassware).
- Any identifying markings (see Section 3.5.2).
- Tolerances (if appropriate, see Section 3.7).
- Measurement conditions:
  - Laboratory air temperature
  - Water temperature(s) at time of test
  - Barometric pressure
  - Relative humidity
- Any out-of-tolerance conditions.

The units reported on the calibration certificate shall match the units marked on the glassware. This requirement is related to ASTM E694 Section 3.12 [12], which does not allow scales or graduation lines for two different measurement units (e.g., milliliters and fluid ounces).

The calibration certificate shall only certify the parameters that were inspected or calibrated. For example, ASTM E288 has several requirements for volumetric flasks, but typically some of these are ensured by the manufacturer and not checked by calibration labs beyond inspection for damage. If the calibration process only determined the volume, the calibration certificate should not state that "the flask is certified to ASTM E288," but rather that "the volume of the flask at 20 °C conforms to the Class A volume specifications of ASTM E288".

If the glassware is not certified to a tolerance, include a suitable statement on the certificate that the uncertainty in use will be higher than the uncertainty stated for the calibration because factors inherent in the use (e.g., uncertainty in reading the meniscus) are additive to the uncertainty reported by the calibration laboratory, and that additional consideration may be needed for any liquids that differ in viscosity and opacity from the liquid used in calibration [16].

3.5.2. Equipment Marking Requirements

Many calibrated items have a new calibration label affixed at each calibration; however, volumetric glassware and any other item that is calibrated gravimetrically (e.g., reference weights) cannot. Not only would a label change the weight, but there is a risk of damage when removing expired labels, and label incompatibility with the cleaning process. The solution is either a unique permanent marking or storage in a marked container with care to keep track of the item when outside of its storage container. A unique marking (e.g., a serial number) provides a means of connecting the item to the calibration certificate and detachable parts that are not interchangeable [12], and distinguishes calibrated from uncalibrated glassware.

Markings are the responsibility of the manufacturer [12], with requirements given in the relevant standards. Note that wide-necked flasks have higher tolerances than standard flasks (see Figure 2 in Section 1.3), leading to a requirement that wide-necked flasks must be marked with their tolerance to avoid confusion [4].

3.6. Environmental, Safety, and Health

Consistent with the approach of ASTM standards (e.g., Section 1.5 of ASTM E542 [18]), this document does not attempt to address all the safety-, environmental-, or health-related hazards associated with glassware calibration. The calibration lab and end user are responsible for determining hazards, establishing appropriate practices, and complying with regulations (see Section 1.4: The Need for Critical Thinking). A few potential hazards are, however, highlighted:

- Reference [16] notes that "Volumetric glassware should not be emptied by holding onto the neck alone. The bottom of the flask should always be supported to prevent glassware breakage and possible injury."
- The cleaning processes have the greatest potential for chemical hazards in the calibration lab.
- All items delivered to the calibration lab must be decontaminated prior to delivery (see Section 3.2).
- Depending on workload and number of repeated measurements, calibration processes may cause ergonomic strain.

3.7. Glassware Accuracy Classes

The ASTM standards have two accuracy classes[24]: Class A is precision grade and Class B is general-purpose grade. Unless a standard specifies otherwise, the volumetric tolerances for ASTM Class B glassware are twice the tolerances for Class A [12].

The materials for Class A and Class B are different [20], with Class A being constructed of a low-expansion borosilicate glass, and Class B being constructed of an alumino-borosilicate glass. The specification for linear coefficient of expansion[25] for the Class A glass makes this class less sensitive to temperature changes and is a key reason why the specified tolerances can be achieved. Note that performing a calibration measurement with lower measurement uncertainty cannot turn Class B glassware into Class A. The same principle applies to accuracy classes from other standards bodies, such as ISO.

3.7.1. Tolerances

The source document for the tolerances that apply to a given item is the relevant ASTM, ISO, or other standard. While NISTIR 7383 [17] provides many details for calibration, it does not provide tolerances. The relevant standards provide tolerances for specific capacities, and if intermediate capacities are possible, the same document will provide the rule for determining those tolerances. The specific standard should always be consulted because the rule for intermediate capacities may not be the same for all types of apparatus[26]. For example, ASTM E287 Section 1.1.3 allows unusual size burets to be Class A if they conform to the tolerance of the next *smaller* size [9], which contrasts

[24] ISO has its own accuracy classes and while the principles in this document apply to all standards systems, specific example values used as examples will be taken from the ASTM standards only.
[25] ASTM E438 [20] specifies the linear coefficient of expansion for the glass, however, when calibrating glassware for volume the correction to the reference temperature (20 °C) is calculated using the coefficient of cubical expansion (CCE), which is 3 times the linear coefficient. Care must be taken to ensure the correct coefficient is used. Values for the CCE are given in ASTM E542 [18] as 0.000010 $cm^3/°C$ for Class A and 0.000015 $cm^3/°C$ for Class B.
[26] In determining appropriate tolerances for custom glassware, the calibration lab may also need to consult the appendix of ASTM E694 [12], which discusses the relationship between the internal diameter of the vessel at the meniscus and the limits of volumetric error.

with ASTM E288 Section 1.1.3 that allows special-size flasks to be Class A if they conform the specifications for the next *largest* size volumetric flask [4].

A tolerance, limit of error, or maximum permissible error (MPE) should be interpreted as the performance expected in normal conditions of use; i.e., in an appropriate laboratory environment, with properly trained or skilled personnel. Manufacturer specifications are often smaller ranges than the tolerances specified in the standards, which may reflect differences in the underlying assumptions, or the influence of marketing practices. Users should default to the tolerances specified in the relevant standards. An example of how this recommendation can be applied for the calibration of POVA-type pipettes is as follows:

1. When the pipette is new, it is adjusted to meet the manufacturer specifications (which are more stringent than the corresponding ISO 8655 tolerances). Calibration data demonstrates that, as left, the pipette meets the manufacturer specifications at the time of calibration.
2. With customer agreement on the assigned calibration interval, the calibration lab can then certify that the pipette is expected to meet the ISO 8655 tolerances over the assigned interval. The difference between manufacturer specifications and ISO 8655 tolerances provides some allowance for drift or degradation in the performance of the device over time.
3. At the end of the calibration interval, the calibration lab performs the as-found calibration (prior to any adjustment or preventive maintenance) and determines the as-found pass or fail condition using the ISO 8655 tolerances that were certified over the interval.
4. Preventive maintenance (cleaning, lubrication, etc.) is then performed and, if necessary, the pipette is adjusted to meet manufacturer specifications. Iterative measurement and adjustment may be performed without recording these results.
5. The as-left calibration is then performed to provide post-adjustment data showing that the pipette meets the more stringent manufacturer specifications. This ensures that the pipette starts the next calibration interval in the best condition practically achievable.
6. The new calibration certificate states that the pipette is expected to meet the ISO 8655 tolerances over the assigned interval, again allowing for some drift or degradation in performance over time.
7. The process repeats from Step 3 until the device is retired with a closeout calibration. Over the life of the device, the calibration interval may be adjusted as appropriate for the intended use. Shorter intervals can be specified, or early calibration can be requested for more severe use conditions or other factors.

For a volumetric apparatus that can measure multiple volumes, the tolerance applies at any graduation line or volume unless the applicable standard states otherwise. This is stated in many standards, such as ASTM E694, Section 3.4, using the example that on a 100 mL graduated cylinder with a limit of error of ± 1.00 mL, the volume at 10 mL could range from 9.00 mL to 11.00 mL [12]. Thus, what is only a 1 % relative error at full capacity is a 10 % relative error at 10 % of maximum capacity. There is usually an accuracy benefit when using the smallest apparatus with sufficient capacity for the work. The same observation is made in connection with Figure 2 for piston-operated pipettes.

3.7.2. The Relationship Between Tolerances and Measurement Uncertainty

If (as is common) a tolerance is assigned to an item, the calibration certificate is required to state both the tolerance assigned and the measurement uncertainty of the calibration. These are different

quantities and must not be confused. This requires clarity in the design and wording of the calibration certificate.

As stated above, the tolerance represents the performance that a device can be expected to achieve in normal conditions of use. The performance achievable in the use of glassware is never the same as the uncertainty in calibration because some of the same contributors (e.g., reading the meniscus) occur in both calibration and use. The uncertainty contributors related to use of the glassware are additive to those reported by the calibration laboratory [16].

A widely used practice when calibrating items and determining compliance with a tolerance is to ensure that the uncertainty of the calibration process (expressed at a 95 % level of confidence) is less than or equal to some fraction of the tolerance being tested. When this approach is used within the DOE, this fraction is 1/4 or 25 % [2]. If this ratio cannot be met, there are options such as guardbanding, where provided the tolerance remains greater than 1.5 times the uncertainty, the limits that must be met are tightened when making the compliance decision [21]. There are various formulas to determine how guardbanding is done, depending on the risk-management requirements[27]. The goal is to control the "risk of false accept", or the risk of concluding that something meets a tolerance it potentially may not. There are other methods of controlling risk, and other fractions in use elsewhere[28], but for all approaches the uncertainty of the calibration process should be less than the tolerance being tested, ideally significantly less.

ASTM E542 [18] makes recommendations for measuring equipment that should achieve the necessary uncertainty in calibration, as does SOP 14 [17].

Since uncertainty is never zero, if a calibration measurement is close to the limits of a tolerance, the range of values included in the uncertainty (at a confidence level of 95 %) may in some cases straddle the limit (see Figure 3). In these cases, the question arises of whether to call the result a pass or fail. The rule that a calibration laboratory follows to determine how to account for uncertainty in this situation is called a decision rule. ISO/IEC 17025 requires that if a calibration certificate makes a compliance statement (e.g., compliance with a tolerance), the decision rule must also be documented.

ISO/IEC 17025 requires that unless a decision rule is inherent in the standard being used, the decision rule be clearly communicated to and agreed upon with the customer [13]. Generally, when the measurement uncertainty is a sufficiently small fraction of the tolerance, the PSL uses the "simple acceptance" decision rule, where the pass/fail decision is made by comparing the measured value directly to the limits. The transparent reporting of the measurement results, their uncertainty (at a stated level of confidence), the assigned tolerance, and the decision rule are necessary for the customer to make their own assessment of the level of false-accept risk.

[27] Organizations within the DOE may contact the PSL if guidance is required on this topic.
[28] GMP 12 in Reference [14] uses a criterion of uncertainty being less than 1/3 of the tolerance when selecting calibration procedures for mass calibrations.

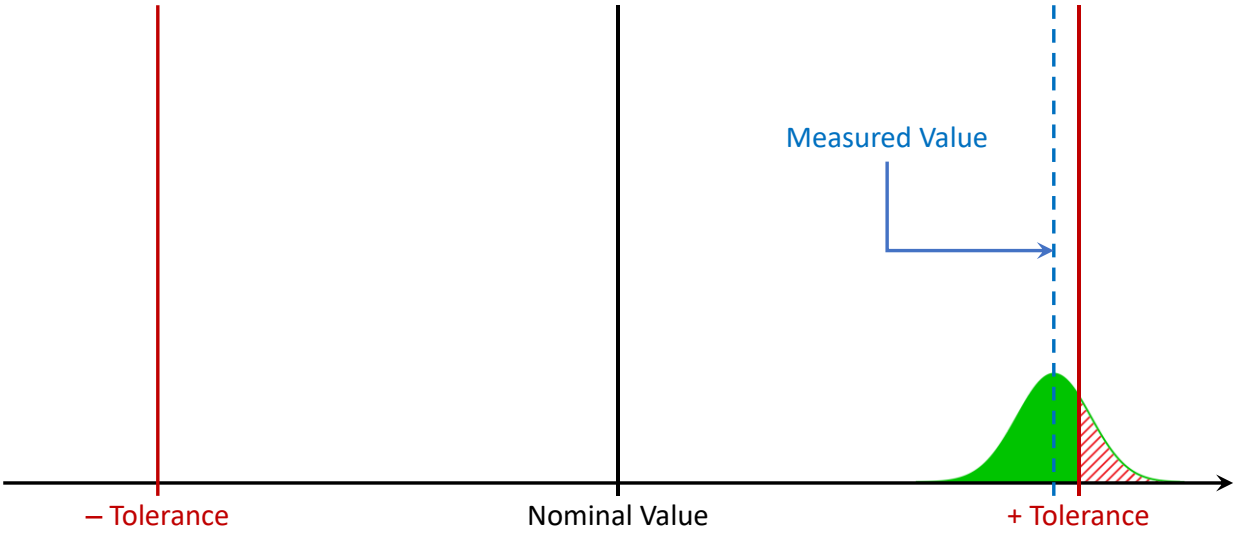

Figure 3. A situation requiring a decision rule for determining compliance with a tolerance or specification: The green bell curve represents the measurement uncertainty in the measured value. The red hatched area represents the probability that the measured value lies outside the positive tolerance (the risk of false acceptance).

3.7.3. Glassware That Does Not Meet Tolerances

While it is theoretically possible to assign a correction factor to glassware that does not meet the class tolerances that correspond to its markings, this is strongly discouraged. New glassware that does not meet tolerances should be discovered at the initial calibration before it is placed in service and returned to the manufacturer for replacement. Used glassware that has changed and no longer meets tolerances should be retired and replaced. Assigning a correction factor relies on the end user remembering to apply the correction factor and doing it correctly. The question should be asked whether the potential consequences of using out-of-tolerance items are so insignificant that replacing the glassware cannot be justified.

3.8. Contaminated Glassware (Radioactive or Other Contamination)

Glassware for volume measurement in an application where decontamination is difficult or impossible (e.g., radioactive contamination) must be calibrated before use, which is straightforward since new glassware is not contaminated. However, a unique problem arises when the glassware is due for periodic recalibration or closeout calibration because the contamination or potential contamination prevents it from returning to the calibration lab. Without these latter calibrations, it is unknown whether the glassware stayed within tolerance throughout the calibration interval.

Calibrating the glassware inside the contaminated area might appear to be a solution, but the calibration standards used are then also subject to potential contamination, and they also require periodic recalibration and closeout calibration, meaning the problem is simply transferred to another item.

The ideal solution is if the contaminated glassware can be calibrated gravimetrically in the laboratory where they reside[29] with a process that ensures the required mass standards are not subject to

[29] Within the DOE any lab that desires to perform its own calibration work will need authorization to do so from the CSL at that site.

contamination. If this is possible, the problem is avoided, but if it cannot, another approach is needed.

A possible approach is to calibrate new (uncontaminated) items and introduce them to the contaminated area. The items already in the area that are due for recalibration are then compared to (or calibrated against) the new, freshly calibrated items. This approach could be implemented two ways, with both ways requiring some means (e.g., an analytical balance) inside the contaminated area to perform the comparison:

1. If there is only one item or a small number of items to be recalibrated, it may make sense to directly replace the items with like items.
2. If there are many items (glassware) to recalibrate, replacing them all may be more expensive than replacing a set of calibration standards (reference weights) used for recalibrating all the items.

The challenge with both approaches is in meeting the requirement for calibration uncertainty to be less that the tolerance assigned to an item (see Section 3.7.2). If the existing item has a tolerance of X, and you replace it with a like item, ideally it should be introduced to the contaminated area after being calibrated to a tolerance that is some fraction of X, X/C, where C is some constant. This would give the comparison (or calibration) a low false accept risk when determining whether the existing item is still within tolerance, but as the process repeats, the item due for replacement can only be checked to the X tolerance instead of the X/C tolerance. The whole scheme rests on the assumption that an item freshly calibrated is very unlikely to be out of tolerance, which is not a bad assumption, but still an assumption.

There is a trade-off in costs not only in the number of items that need to be periodically replaced but in the required calibration equipment. If the solution involves replacing glassware, the required analytical balance will need resolution and measurement uncertainty suitable for glassware calibration, whereas if replacing weight sets, the required balance will need higher resolution and lower uncertainty suitable for weight calibration and will undoubtedly cost more.

The risks of not detecting an out-of-tolerance condition should be considered and managed appropriately. Methods like increasing the number of repeated measurements in the calibration process and using control charts for check standards can manage and minimize the uncertainties and risks.

Even with careful consideration of costs and risks, generating "waste" may be unavoidable. The process should not, however, be considered wasteful, but rather the cost of ensuring measurement quality in a contaminated environment.

3.9. Standardize Before Use

If the conventional calibration process is not feasible (e.g., due to contamination, destruction of the glassware during use, or other considerations) you can verify the accuracy of the glassware at the time of use. At Sandia National Laboratories this process is called Standardize Before Use (SBU). The word "calibration" is not used because no calibration certificate and no calibration label are produced.

As the goal of the SBU process is traceability, the same seven essential elements discussed above are applicable, but may have to be modified due to the constraints that prevent conventional calibration. At Sandia National Laboratories, assets designated as SBU are required to have a written

standardization procedure, personnel approved to perform the procedure, PSL-certified standards traceable to the SI, etc. In lieu of calibration certificates, other records (such as laboratory notebooks) are kept to document standardization activities and results. An uncertainty analysis is also required at a level of rigor commensurate with the application.

At DOE sites, SBU activities are approved by the CSL for that site.

3.10. What About Plastic?

The calibration procedures for volumetric glassware (SOP 14 [17] and ASTM E542 [18]) are applicable to volumetric containers made from other materials such as metal or plastic, provided the material of construction is stable and a value for the cubical coefficient of thermal expansion is available. Table X1.1 in ASTM E542 provides thermal coefficient of cubical expansion (CCE) values for six different plastics commonly used for laboratory ware.

The key differences in calibrating plastic volumetric apparatus relative to glass are the achievable accuracy, calibration intervals, and cleaning processes.

As noted in Section 3.7, the cubical coefficient of thermal expansion is a factor in achievable accuracy in normal use and the reason why precision grade (Class A) glassware is made from low-expansion glass. The CCE values for plastics given in ASTM E542 range from 5.5 to 45 times higher than the value for Class A glassware and 3.67 to 30 times higher than the value for Class B glassware. There are other specifications in the standards that could directly affect the achievable accuracy as well, such as thickness of graduation lines and maximum inside diameter at the capacity line. The meniscus will also exhibit some differences relative to glass due to the hydrophobic nature of a clean and smooth plastic surface. It is unreasonable to expect that plastic volumetric apparatus could achieve the same tolerances as glass apparatus; however, a specific application may require plastic for material compatibility reasons. If an evaluation of achievable accuracy indicates that the apparatus cannot achieve the required accuracy (see Section 1.3), plasticware can be used to contain the materials, but you will need to make measurements gravimetrically instead of volumetrically.

The discussion on calibration intervals and the possibility of calibration intervals as long as 5 years (see Section 2.3) is based on experience with glassware. In the absence of stability data specific to the type of plastic and use conditions, plasticware should be calibrated annually until sufficient data is accumulated to justify longer intervals.

The cleaning methods recommended for glassware may not be compatible with some or all plastics. SOP 14 recommends following manufacturer instructions for cleaning and not using cleaning agents that can attack, discolor, or swell the material [17].

3.11. POVA-Type Pipettes

Unlike volumetric glassware, piston-operated pipettes have moving parts and seals that require periodic maintenance, such as cleaning and lubrication, and possibly replacement. Calibration intervals longer than one year are not recommended for these devices. Preventive maintenance should be performed at every calibration, but only after as-found calibration data is collected.

More frequent calibration may be appropriate, depending on the conditions of use. Conditions that may justify more frequent calibration include:

- High frequency of use
- Large number of operators

- Large number of dispensings performed during each use[30]
- Corrosive or solvent nature of the liquid dispensed

Routine testing between calibrations with a risk-based approach is recommended and can help avoid rework. This process is the responsibility of the end user. Refer to ISO 8655:2022 Part 7 for details [22].

The MPE values in ISO 8655-2:2022 apply to the pipette and tip as a system. The values stated in the ISO standard represent performance that can be realistically achieved, whereas manufacturer specifications may assume specific tips are used or other qualifying assumptions [22]. See Section 3.7.1 for a calibration process example where both manufacturer specifications and ISO standard MPE values are used to good effect.

Calibration is performed gravimetrically in a manner that is consistent with the definition of the assigned tolerances. For example, ISO 8655 defines the maximum allowable systematic error as the deviation of the mean of a tenfold measurement, and the maximum permissible random error as the standard deviation of a tenfold measurement. The calibration procedure to check these devices to the ISO 8655 standard should therefore involve a tenfold measurement at each volume calibrated.

3.12. Ethics

The results of calibrations affect the quality of goods and services provided using the calibrated items, and the quality of these goods and services can potentially impact public safety. Because of this connection, ethics and calibration are inseparable. Calibration personnel have an obligation to the public to adhere to the highest standards of integrity. Calibration-specific examples where personnel can act with integrity include:

- Calibration data shall be reported completely and with objectivity. Selective reporting of data to achieve a specific outcome is never appropriate. Outliers in data should be identified by objective statistical methods rather than opinion. Check standard data should not be excluded from a control chart unless there is an assignable cause for it being an unacceptable data point.
- Decisions (e.g., adjustments to calibration intervals) should be documented and based on data and sound engineering judgement, with appropriate consideration for risk, particularly to the safety of the public.
- Results shall be reported with transparency. For example, reporting results with the associated measurement uncertainty and any decision rule applied allow the user to assess the false-accept risk.
- Users that are impacted by out-of-tolerance conditions shall be notified. Users are notified by the calibration certificate when their own items are found outside of certified tolerances, but potentially affected customers should also be notified when the calibration laboratory's equipment or standards are found out of tolerance.
- Any damage or events that may impact the validity of calibration standards or the accuracy of M&TE shall be immediately reported so the potential impact can be quantified and mitigated. To maintain a safety-conscious work environment, it is critical that laboratory

[30] It is permissible to base expiration of calibration intervals on criteria other than time, such as number of uses. This does require a means to track the number of uses, and if this approach is used should be combined with a maximum time for example, "100 uses or 1/31/2025, whichever comes first".

supervisors welcome such reports without retaliation or consequences for the reporting employee that would discourage such reporting.

Calibration results and the associated uncertainty information that are ethically produced are complete and defensible.

4. CONCLUSION

This document provides metrological guidance for the calibration of laboratory volumetric glassware discussed in the context of NIST's seven essential elements of metrological traceability and other considerations. While some requirements are inflexible, the end user has many options. It is the intent of this document to allow options whenever possible, with recommendations to which options may be most beneficial. The following is a summary of requirements that must be followed to achieve metrological traceability.

- If a measurement can affect the quality of a product or service, the M&TE (e.g., volumetric glassware) shall be calibrated.
- The quantity being measured shall be fully defined in terms of SI units or accepted conversions from SI units.
- Calibration results shall be traceable through an unbroken chain of calibrations to accepted national, international, or intrinsic standards.
- Calibrated items shall be recalibrated at appropriate intervals and calibrated prior to retirement. (Challenges associated with calibration of contaminated items are acknowledged).
- All links in a traceability chain shall have documented measurement uncertainty determined by accepted methods and reported at a stated level of confidence.
- Calibrations shall be performed using documented and validated procedures.
- The calibration laboratory shall provide evidence of metrological traceability.
- The calibration laboratory shall have a measurement assurance program.
- Customers shall ensure glassware is free of contamination (biological, chemical, radiological, etc.) before delivering glassware to the calibration lab.
- Calibration certificates shall include all information required by ISO/IEC 17025 and additional information specific to glassware calibrations specified in this document (see Section 3.5.1).
- Calibration certificates for outsourced calibration work shall be carefully reviewed by an SME to assess the validity of results and evidence of metrological traceability before acceptance.
- Calibrated items shall be marked or otherwise uniquely connected to their calibration documentation.
- Calibration laboratories shall document and communicate the decision rule when determining compliance to a tolerance or standard.
- DOE entities performing verification of glassware at the time of use (SBU) shall obtain approval of the process from the CSL at their site.

The following is a summary of major recommendations.

- If volumetric glassware cannot meet the process requirements, the process should be redefined to use gravimetric measurements instead of volumetric measurements.
- For the lowest measurement uncertainty, the smallest volumetric apparatus capable of performing the function should be employed.
- Users and calibrators of volumetric glassware should become familiar with the relevant published standards and take care to apply critical thinking.

- Items should be calibrated in a manner that is consistent with their use.
- Any factors that affect the calibrated value should be measured, controlled if possible, and their values reported with the calibration data.
- Traceability chains should be kept as short as practical to minimize measurement uncertainty.
- When selecting calibration intervals, consider the conditions of use.
- Do not attempt to justify excessively long calibration intervals to avoid calibration. This is a self-defeating effort, since a strong justification requires data that calibration provides.
- Adopt published and validated procedures such as ASTM E542 or SOP 14 without modification, and supplement with local procedures to provide any needed specifics.
- Laboratory glassware should be calibrated by gravimetric methods rather than volume transfer methods.
- Calibration laboratories control the quality of water with treatment processes rather than measure the density of an uncontrolled water source, as uncontrolled water sources may contaminate the glassware.
- Calibration laboratories and users should coordinate to ensure compatibility of cleaning chemicals with both calibration and use processes.
- Calibration laboratories should pay careful attention to the work area layout to minimize variation in temperature across the work area and to maximize ergonomics.
- Formal processes (e.g., forms) should be used when communicating with the customer.
- When new glassware does not meet published tolerances, return it to the manufacturer rather than place it in service with a correction factor.
- Assess costs and measurement risk when determining how to implement a calibration program for glassware with radioactive contamination.
- Calibration intervals for volumetric apparatus with moving parts and seals should not exceed one year.

For practitioners within the DOE, the PSL is available to provide measurement and calibration guidance to supplement and clarify the information herein.

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