Title: Guidance on Traceability of Measurement

TABLE OF CONTENTS

1.0 SCOPE

The AIHA-LAP, LLC Traceability of Measurement Policy documents the requirements for accredited laboratories to maintain accreditation to ISO/IEC 17025 with regard to traceability of measurement. This guidance document provides additional information intended to assist laboratories implement the Policy efficiently and effectively. AIHA- LAP, LLC wishes to thank and acknowledge the Canadian Association for Laboratory Accreditation (CALA) for its permission to incorporate elements of CALA A61-01 Policy and A61-02 – *CALA Traceability Policy and Guidance, respectively)* in preparing this guidance document.

2.0 REFERENCES

The following documents, or future versions thereof, provide the basis and assist with application of the principles stated in this policy.

- **AIHA-LAP, LLC Policy Appendix G on the Estimation of Uncertainty of Measurement**
- **AIHA-LAP, LLC Guidance on the Estimation of Uncertainty of Measurement**
- **AIHA-LAP. LLC Policy Appendix H Traceability of Measurement**
- **CALA Example Internal Calibration Workbook**
- **CALA Traceability Policy & Guidance on Traceability,** www.cala.ca
- **General requirements for the competence of testing and calibration laboratories,** ISO/IEC 17025:2017
- **Guidelines for the determination of calibration intervals of measuring equipment**, ILAC G24, www.ilac.org
- **ILAC Policy on the Traceability of Measurement Results,** ILAC P10, www.ilac.org
- **NISTIR 6919 – Recommended Guide for Determining and Reporting Uncertainties for Balances and Scale**
- **NIST Special Publication 1088- Maintenance, Validation, and Recalibration of Liquid-in-Glass Thermometers**

3.0 TERMS AND DEFINITIONS

Refer to the AIHA-LAP, LLC Traceability of Measurement Policy and Policy and Guidance on the Estimation of Uncertainty of Measurement for applicable definitions.

4.0 BACKGROUND

The guidance provided in this document is intended as helpful suggestions. It is important to note that it is not the place of accreditation bodies to make business decisions for their member laboratories. Rather, it is the responsibility of each individual laboratory to choose to implement any or none of the methods described in this document, based on its individual needs and its individual assessment of risks, as long as the AIHA-LAP, LLC Traceability of Measurement Policy is met. It is also the responsibility of the laboratory to evaluate the effectiveness of the method it chooses to implement and take responsibility for the consequences of the decisions taken as a result of the method chosen.

AIHA-LAP, LLC accredited laboratories must understand the following simple relationship. All three of these components must exist at every level in the traceability chain in order for the final test result to be traceable.

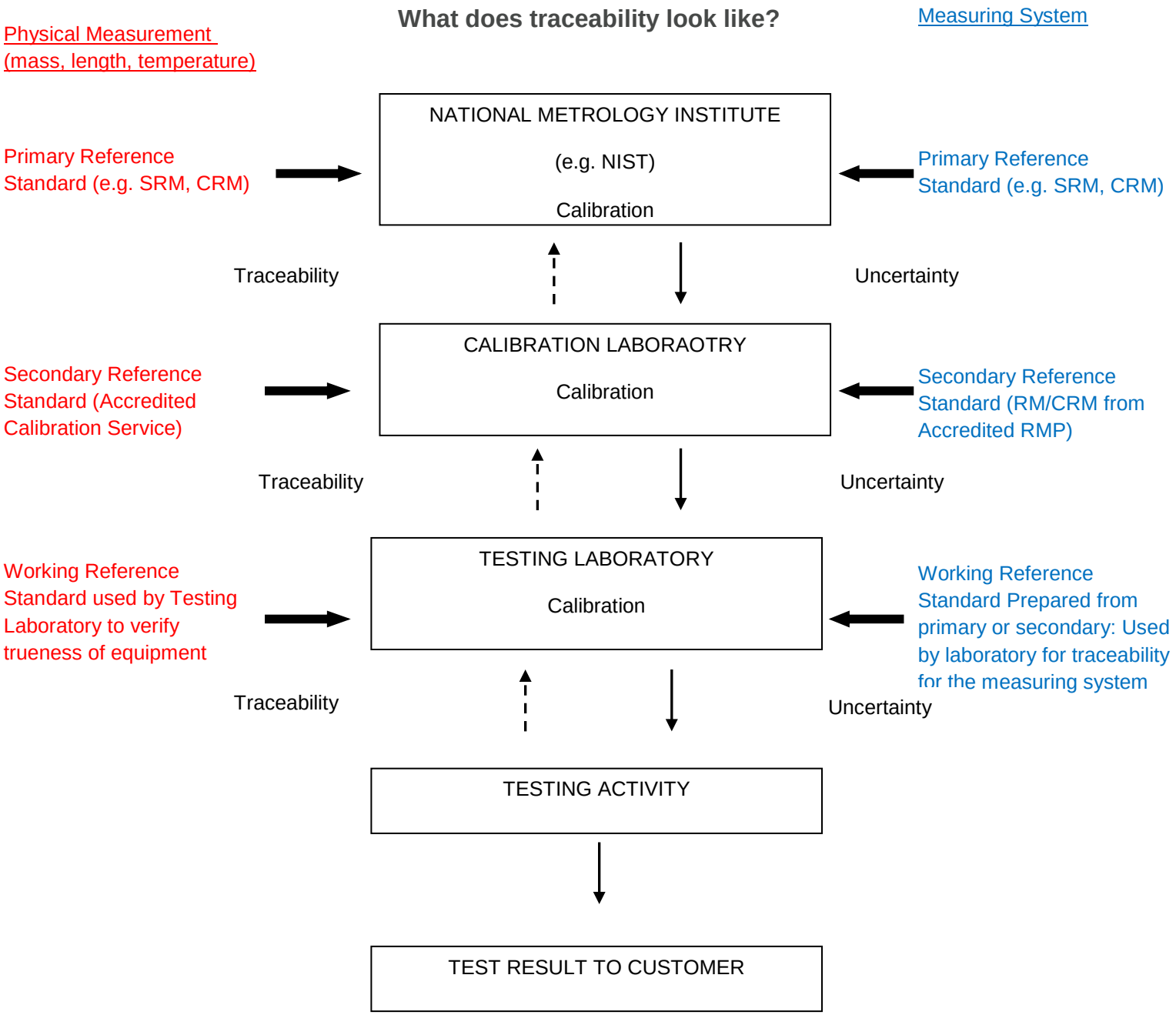
(1) (2) (3)

Calibration with Uncertainty produces a measurement result that is Traceable

The following figure and description will hopefully assist in understanding some of the terms and relationships related to traceability.

Figure 4-1

What does traceability look like?



At the top of this diagram are all of the National Metrology Institutes (NMIs) that are responsible in each nation for quantifying specific parameters for use within their nation. Most NMIs have signed a multilateral recognition agreement based on the "demonstration of competence" (e.g., accreditation against ISO/IEC 17025).

The first job of an NMI is to characterize a parameter, such as mass, temperature, or mass per volume to a specific level of uncertainty. They estimate this uncertainty in support of traceability of the measurements. This affects legally mandated measurements (butcher weigh scales and gas pumps) as well as other fields of science requiring competent measurement. This is required in order to ensure that measurements are comparable throughout the world.

Each NMI has the ability to conduct measurements with very small uncertainties. Uncertainties increase from the primary reference standard to secondary reference standards and finally to the testing laboratory. NMIs are also able to calibrate instruments or establish quantity values of certified reference materials to establish traceability to the SI.

5.0 GUIDANCE FOR IMPLEMENTATION

Laboratories need to make several decisions when establishing their calibration and verification procedures and frequencies. The following will be discussed in turn:

- Which equipment needs to be calibrated?
- When are verifications needed?
- At what frequency should both occur?

1) Which equipment needs to be calibrated?

ILAC identifies all equipment that can impact the measurement uncertainty of test results as "critical" equipment and considers it in need of calibration. Recall that calibration involves traceability and uncertainty. So laboratories will either have to purchase external calibration services or perform in-house calibrations to obtain the traceability and measurement uncertainty for critical equipment. The process for estimating measurement uncertainty for a calibration follows the same policies, principles, and calculations already defined in the AIHA-LAP, LLC Policy on the Estimation of Uncertainty of Measurement and its related guidance document.

2) When are verifications needed?

Verifications are used to indicate whether or not the equipment is still in calibration. They should be performed as often as needed to provide the laboratory and its customers with confidence in the calibration status of the laboratory's equipment.

3) At what frequencies should calibrations and verifications occur?

There is no one answer as each laboratory must consider the testing it performs, its customer's needs and various aspects of its operation. ILAC provides the following list of considerations:

- uncertainty of measurement required or declared by the laboratory;
- risk of a measuring instrument exceeding the limits of the maximum permissible error when in use;
- cost of necessary correction measures when it is found that the instrument was not appropriate over a long period of time;
- type of instrument;
- tendency to wear and drift;
- manufacturer's recommendation;
- extent and severity of use;
- environmental conditions (climatic conditions, vibration, ionizing radiation, etc.);
- trend data obtained from previous calibration records;
- recorded history of maintenance and servicing;
- frequency of cross-checking against other reference standards or measuring devices;
- frequency and quality of intermediate checks in the meantime;
- transportation arrangements and risk; and
- degree to which the servicing personnel are trained.

The Table below contains minimum calibration and verification frequencies. It is imperative laboratories understand that this table is not a list of recommended frequencies. Laboratories that rely only on the minimum frequencies may be compromising their test results.

Common Reference Standards and Support Equipment Requiring Calibration and/or Verification

Reference Standard / Equipment	Calibration Frequency	Verification Frequency
Reference Thermometer	Initial and as determined by the laboratory	Not applicable
Working Thermometer	Not Applicable	As defined by the laboratory
Reference Masses	Initial and as determined by the laboratory	Not applicable
Working Masses	Not applicable	As defined by the laboratory
Stage Micrometer	Initial and as determined by the laboratory	As defined by the laboratory
Balance	Initial and as determined by the laboratory	As defined by the laboratory
Mechanical Pipettes	Initial and as determined by the laboratory	As defined by the laboratory
Volumetric Containers for critical functions	Initial and as determined by the laboratory	As defined by the laboratory

5.1 EXTERNAL CALIBRATIONS:

5.1.1 Purchasing Reference Standards

Reference standards can be readily purchased with a calibration certificate that demonstrates the calibration was performed by an ISO/IEC 17025 accredited calibration laboratory. Reference standards can include certified thermometers, weights, stage micrometers, etc.

Reference standards must be accompanied by a calibration certificate. Refer to 5.1.3 below. Refer to the AIHA-LAP, LLC Traceability of Measurement Policy for the minimum recalibration or replacement frequency.

5.1.2 Purchasing Calibration Services

Many calibration laboratories have been accredited to ISO/IEC 17025 for their services. When choosing a calibration laboratory, obtain their scope of accreditation and ensure they are accredited for the service you are hiring them for, for example, calibrating a balance, mechanical pipette or thermometer in the operating range you need.

Maintain a copy of their scope of accreditation as part of your service provider evaluation records (ISO/IEC 17025 section 6.6.2 d).

The service provider will provide a calibration certificate that will include several pieces of information, as listed in ISO/IEC 17025 section 7.8. It is important to review the certificate to determine if the “as found” values could have impacted test results reported since the last calibrations and if the measurement uncertainty of the equipment continues to support the testing activities.

5.1.3 Calibration Certificates

Certificates accompanying reference standards or provided in support of other calibration services are traceable to the equipment via serial numbers. The certificates must be kept for the life of the reference standards or equipment, and maintained according to ISO/IEC 17025

The following will appear on an ISO/IEC 17025 compliant calibration certificate for reference standards and support equipment:

- A statement that the calibration laboratory is accredited to ISO/IEC 17025. This statement may be supported by a recognized accreditation body symbol or the symbol may appear by itself.

- The serial number of the measuring equipment/reference standard being used to calibrate your reference standard or equipment and a statement that the measuring equipment is traceable to SI units through an NMI.

- The measurement range for which your equipment or reference standard was calibrated and the specific uncertainty measurements for that range.

If these are not on the Calibration Certificate, then the Certificate will not meet the requirements of ISO/IEC 17025 and the AIHA-LAP,LLC Traceability of Measurement Policy.

Before purchasing a reference standard or calibration service, you may wish to request an example of their calibration certificate. This will allow you to address any concerns you have with your supplier before you purchase the item or service.

Also note the cost for traceability is usually higher than the non-traceable calibration, so if you are choosing between levels of service and are paying the lowest price for a calibration or reference standard, it's probably not traceable.

5.2 IN-HOUSE CALIBRATIONS

Calibrations are considered to be performed in-house if personnel performing the calibrations and verifications have received documented training and are performing the calibrations and verifications in accordance with the same ISO/IEC 17025 compliant laboratory quality management system that applies to other aspects of its analytical operations covered by its AIHA-LAP, LLC accreditation. Calibrations performed by sections of the organization operating under a different quality management system are not considered in-house.

Frequencies and tolerances must be defined for all calibrations performed in-house. The tolerances determine when the equipment no longer meets the maximum error or bias or precision requirements of the intended use.

5.2.1 Measuring Systems Using Analytical Instruments

Measuring systems use a variety of analytical instruments and support equipment; including one or more of the following: balances, pipettes, chromatographs, spectrographs, volumetric ware, etc., to generate measurement (test) results. Each of these instruments and support equipment may contribute to the overall uncertainty of the measurement result and require calibration and traceability.

A laboratory performing calibrations of the measuring system (organic, inorganic, and microbiological measurements) must follow a documented instrument calibration procedure as defined by a reference method, regulatory standard, other consensus testing methods, or laboratory standard operation procedures and utilize reference materials that satisfy traceability requirements in accordance with Sections 5.3 and 5.4 of the AIHA-LAP, LLC Traceability of Measurement Policy. The laboratory must maintain certificates of analysis for all reference materials used and maintain sufficiently detailed records to clearly document the specific reference material and associated lot number used for instrument calibration.

The traceability to the SI for chemical and biological measurements is often not achievable. For chemical measurements the ideal is the traceability to the mole and for biological measurements no SI has been defined. For microbiological measuring systems, the use of reference cultures (materials) from an accredited or recognized microbiological reference material producer is the best practice for traceability of the measuring system. For chemical measuring systems, the use of reference material from accredited reference material producers or NMIs, when possible, is the best practice for traceability of the measuring system. With over 10,000 possible chemical and microbiological measurands, reference standards from accredited producers are not always available. Refer to Section 5.6 of this guidance document for information regarding selection of a supplier of reference materials.

5.2.2 Working / Support Equipment

Working and/or support equipment can include but not be limited to balances, mechanical pipettes, other dispensing devices, and working thermometers, etc. Laboratories may find that in-house calibrations meet some or all of their calibration needs. In-house calibrations also must comply with all measurement traceability requirements including estimating measurement uncertainty from all major contributors. (See ISO/IEC 17025, Section 6.5)

NOTE: *It is important to remember that all internal calibrations must be performed according to a documented procedure by trained staff and that calibration records must be maintained that include all applicable elements of ISO/IEC 17025 Section 7.8.4.*

5.2.2.1 Balances

When using a balance, it must be properly calibrated (traceable with known uncertainty) and it must be demonstrated that its calibration has not changed since the last calibration.

A laboratory may perform its own calibrations as long as it follows an acceptable calibration procedure. NISTIR 6919 – *Recommended Guide for Determining and Reporting Uncertainties for Balances and Scales* allows for the determination of uncertainty and provides useful information.

Calibration involves replicate measurements using traceable reference weights and the estimation of uncertainty from all contributors using the root of the sum of the squares equation. Contributors to measurement uncertainty for this calibration are defined by CALA as:

Uncertainty Contribution Table for Balances			
Contribution (nomenclature)		**Distribution**	**Estimated Value (NISTIR 6919)**
u_s	Uncertainty of the nominal values of the reference weight set	Normal	Expanded uncertainty on the calibration certificate of the weights divided by 2 (coverage factor – **k**)
s_p	Standard deviation of the set of calibration readings	Normal	Standard Deviation of the set of calibration measurements.
U_1	Standard uncertainty associated with the reapeatability of the balance response to the reference weight set	Uniform (Square)	Smallest display increment divided by √3if unable to determine from 7 consecutive readings. **Use only if s_p = 0.**

Refer to the CALA Example Internal Calibration Workbook at:
http://www.aihaaccreditedlabs.org/Policies/Pages/Additional-Resources.aspx

5.2.2.2 Weights (masses)

Weights cannot be calibrated in a laboratory setting. Laboratories must utilize certified weights if they are calibrating their balances in-house. Weights can only be certified or recertified by an ISO/IEC 17025 accredited calibration laboratory according to 5.1above.

However, if the laboratory has used an external calibration provider to calibrate its balances and the weights are used for verification only, then certified weights may not be needed. Refer to section 5.3.2 below.

5.2.2.3 Thermometers

A laboratory may decide to maintain a limited number of thermometers that are sent to an accredited calibration laboratory on a scheduled basis (e.g., every five years). The lab may then use these reference thermometers to calibrate the laboratory's working thermometers. The internal calibration must use an accepted protocol. NIST Special Publication 1088 – *Maintenance, Validation, and Recalibration of Liquid-in-Glass Thermometers* provides useful information.

Calibration of liquid in glass thermometers can be performed in situ at the temperature of use instead of at the ice point. Calibration of digital thermometers is best performed in situ at the temperature of use.

Calibration involves the comparison of replicate measurements using a

reference thermometer and the estimation of uncertainty from all contributors using the root of the sum of the squares equation. Contributors to measurement uncertainty for this calibration are defined by CALA as:

Uncertainty Contribution Table for Thermometers			
Contribution (nomenclature)		**Distribution**	**Estimated Value**
u_r	Standard uncertainty of the nominal values of the reference thermometer.	Normal	Expanded uncertainty on the calibration certificate of the reference thermometer divided by 2 (coverage factor – **k**)
sp	Standard deviation of the set of calibration readings	Normal	Standard deviation of the set of calibration measurements.
u1	Standard uncertainty of the readability and resolution of the working thermometer	Uniform (Square)	Smallest gradation of the working thermometer divided by √3. **Use ONLY if Sp = 0**

Refer to the CALA Example Internal Calibration Workbook at
http://www.aihaaccreditedlabs.org/Policies/Pages/Additional-Resources.aspx

5.2.2.4 Mechanical Pipettes/Dispensers/Dilutors

Dispensing devices may be sent to an accredited calibration laboratory for calibration but this is often cost prohibitive. Alternately, the laboratory may perform an internal
calibration following a generally accepted procedure such as those provided by Troemner, Mettler-Toledo, Eppendorf, and Rainin; refer to their websites. ISO 8655-6 - *Piston-operated volumetric apparatus – Part 6: Gravimetric methods for the determination of measurement error* provides the basis for the contribution table below.

These procedures generally involve the repeated weighing of dispensed volumes of water, corrected for standard temperature and pressure. For adjustable dispensing devices, this procedure is performed at more than one volume. The procedure will generally include 10 measurements at the low end and 10 at the high end of the dispensing range. Acceptable performance (bias and uncertainty) is based on that required for the piece of dispensing equipment. Spectral calibrations methods may also be utilized.

Calibration involves the comparison of replicate measurements using a traceable balance and the estimation of uncertainty from all contributors using the root of the sum of the squares equation. Evaporation loss may be significant depending on the liquid dispensed. Where significant, the replicate data can be corrected for loss due to evaporation. Contributors to measurement uncertainty for this calibration are defined by CALA as:

Uncertainty Contribution Table for Pipettes

Contribution (nomenclature)		Distribution	Estimated Value
u_r	Standard uncertainty of the nominal values of the reference balance.	Normal	Expanded uncertainty on the calibration certificate of the reference balance divided by 2 (coverage factor – **k**)
s_p	Standard deviation of the set of calibration readings	Normal	Standard deviation of the set of calibration measurements.
s_T	Standard deviation of corrections caused by temperature (ΔT) when the temperature differs from standard temperature (20^oC). The thermal coefficient of expansion of water is	Uniform (Square)	Relative Standard Deviation = (ΔT x 0.0002) / ($\sqrt{3}$) in millilitres per millilitre
u_1	Standard uncertainty of the readability and resolution of the working volumetric instrument	Uniform (Square)	Smallest gradation of the working volumetric instrument divided by $\sqrt{3}$. **Use ONLY if Sp = 0**

Refer to the CALA Example Internal Calibration Workbook at http://www.aihaaccreditedlabs.org/Policies/Pages/Additional-Resources.aspx

5.3 VERIFICATIONS

Verifications are performed to demonstrate the equipment is still in calibration. Verifications must have defined frequencies and acceptance criteria that are relevant to the measurement uncertainty of the test methods. For example, the uncertainty of verification weights must be very small as compared to the acceptance criteria of the balance performance it is verifying.

Instruments and equipment that “drift”, or are prone to sudden changes in precision or measurement capability, require periodic verification. Affected measurements include, but may not be limited to, balances, mechanical pipettes, portable thermocouples, and most analytical instruments (spectrometers, chromatographs, etc).

5.3.1 Analytical Instruments

The calibration of such equipment is verified by the routine use of Continuing Calibration Verification (CCV) standards. Analytical procedures or accreditation requirements specify the frequency at which CCVs should be run for each type of system.

5.3.2 Balances

Balances are verified to demonstrate the performance has not changed by using a minimum of two weights that are within the mass range typically measured on the balance. This allows the laboratory to demonstrate stability of operation of the balance over the whole range normally used. If the laboratory restricts use of the balance to a constant single weight measurement, then only that point needs to be verified.

NOTE: The working weights used for verification do not have to be calibrated weights but they must be weighed on a calibrated balance on the same day that the balance calibration is performed. This is not considered a calibration, but rather is a verification that the masses have not changed to a degree that will impact their use for verification in the lab. Tolerances must be established as for all verifications. Working weights can also be verified by comparing them to a set of traceable calibrated weights.

5.3.3 Working Thermometers

Verification of liquid in glass thermometers and digital thermometers are typically performed in situ at the temperature of use through comparison with a calibrated reference thermometer. The verifications are compared with acceptance criteria that include any correction factor applied during calibration.

5.3.4 Mechanical Pipettes/Dispensers/Dilutors

Verifications can be performed by dispensing a measured volume to a tared container on a balance and recording the weight. Verifications can be at the volume of use or at the maximum and minimum volume of variable pipettes – refer to the calibration section for more information. These values are recorded, and acceptance criteria for bias and precision are established, appropriate to the use of the device. For example, if it is used to measure a volume of a standard for preparing calibration standards, its acceptance criteria would be tighter then if it was used to transfer a component of a reagent.

5.3.5 Glassware/Volumetric ware

Class A glassware does not require verification unless over-heated or chipped, for most methods typically used by testing laboratories.

Verification of non-class A glassware and other volumetric ware is needed when it is used for critical measurement (such as disposable graduated digestion tubes). This is easily achieved by sampling each lot of volumetric ware prior to use and verifying it contains the volume needed, either by delivering a known volume into the container and checking against

gradations, or filling to the gradation with distilled water and weighing in the same manner as verifying mechanical pipette calibrations. Acceptance criteria appropriate to the use of the device must be established by the laboratory.

5.4 CALIBRATION AND VERIFICATION RESULTS

The results of calibrations and verifications are examined to determine if the equipment is still in calibration. The "as found" results from external calibrations and the repeated measurement from internal calibrations will provide this information. If they do not meet acceptance requirements, they are treated as a nonconformance and typical corrective action procedures apply.

Corrective action may include, but is not limited to, the re-calibration or replacement of the equipment. The impact on test results since the last verification or calibration must be considered and addressed.

5.5 SELECTING A CALIBRATION LABORATORY

An accredited calibration laboratory or accredited reference material producer is acceptable if the accreditation body is from a recognized ILAC Signatory. This includes recognition through regional cooperation such as APLAC and/or IAAC. The following links are provided for organizations seeking assistance in finding acceptable, accredited calibration laboratories and proficiency test providers. The AIHA-LAP, LLC does not endorse any accredited provider over any other accredited provider

- SCC/CLAS
- NVLAP
- A2LA
- IAS
- ANAB
- PJLA

5.6 SELECTING A REFERENCE MATERIAL PROVIDER

When choosing a supplier of reference materials to be used for traceability of the measuring system, the following factors should be taken into account (adapted from Traceability of Chemical Measurements: Eurachem 2003):

- **Step 1:** Select the best reference material available.
  For the measuring system, identify the reference material needed for traceability. Determine if a primary reference standard is available from NIST or other NMI or if secondary reference standards are available from an accredited reference material producer for chemical and microbiology measurements. An accredited reference material producer (RMP) is a supplier that conforms to ISO 17034in combination with ISO/IEC 17025.. Conformance is demonstrated through accreditation by an ILAC recognized signatory. (Note that reference materials from accredited RMPs are typically available for metals and common inorganic standards and some organic mixtures.)

- **Step 2:** When a primary reference or a reference material from an NMI or from an accredited reference material producer is not available, the laboratory may establish traceability in the following manner:

  a) The track record of both the producer and the material is documented. For example, whether the reference material in use has been subjected to an interlaboratory comparison, cross-checked by use of different methods, or there is experience of use in a number of laboratories over a period of years (certified reference materials (CRM), AIHA-PAT, LLC proficiency testing samples, or other materials with some type of certificate and statement of uncertainty).

  b) There is "Well established purity information for the material." This may apply to neat materials, ACS primary standards, EPA pesticide repository materials, etc.

  c) The laboratory has documentation to show that the material is fit for the intended purpose as agreed to with the customer. The laboratory documentation may be from the reference method or a regulatory or customer requirement requiring the use of a given reference material (including customer supplied materials such as solvent mixtures, pharmaceuticals, mineral/cutting oils, etc.).

An accredited reference material producer is acceptable if the accreditation body is from a recognized ILAC Signatory. This includes recognition through regional cooperation's such as APLAC and/or IAAC. The following websites present some of the sites to find accredited reference material producers:

- SCC/CLAS
- NVLAP
- A2LA
- ANAB

Primary reference standards and secondary reference standards may also be obtained from an NMI. Visit the NIST website for information describing available standard reference materials. The following website presents information on available primary reference standards for chemicals: http://www.aqc.it/en/ and www.bipm.org.