A61-02 – CALA GUIDANCE ON TRACEABILITY

Revision 1.3

December 21, 2018

CALA
Trust, measured accurately

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GUIDANCE

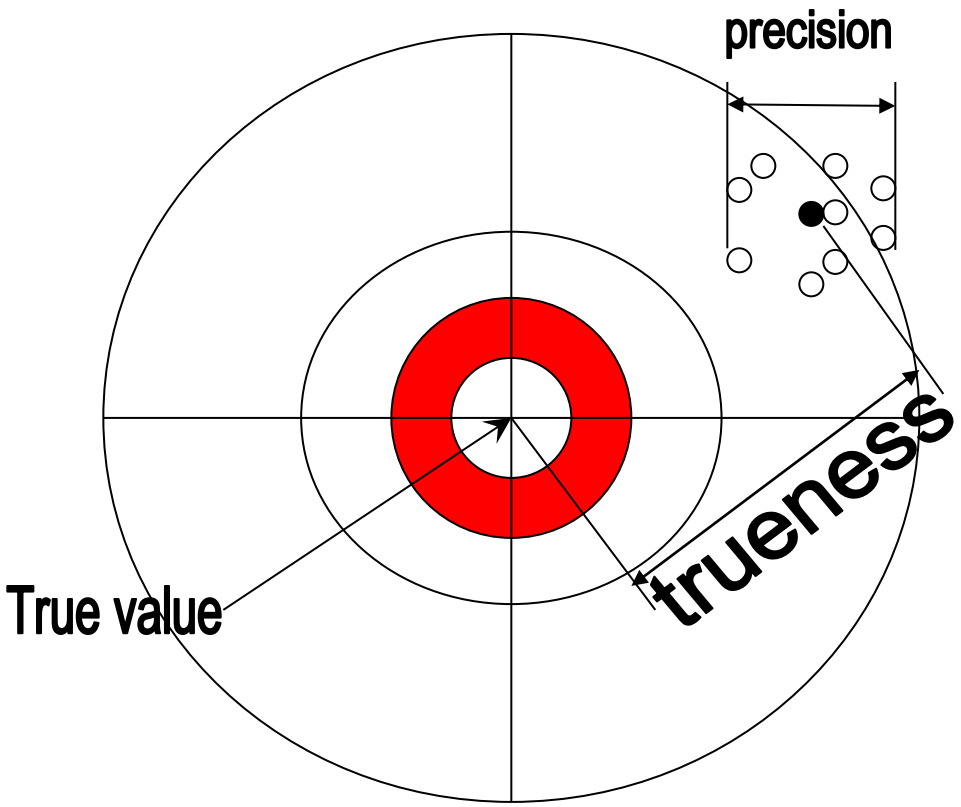
1.0 INTRODUCTION

This document is provided for guidance on the implementation of A61-01 CALA *Traceability Policy*. It is not a requirements document.

2.0 UNCERTAINTY AND TRACEABILITY

Traceability is related to the concept of Uncertainty of measurement.

This diagram provides demonstration of their relation.



This diagram shows a series of results as the small white dots. They are not the same as the actual value one would get if we lived in a perfect world. This perfect or true value is represented by the centre of the target.

The black dot represents the average of all the results generated. It is not a result itself. It is also called the *mean* or *average* of the set of generated results. If a laboratory has produced a set of results from one large sample, then they may report this average as representative of the whole set.

Whether a laboratory reports a single result or the mean (average) of a set of results, the considerations for reporting uncertainty are the same in all cases:

- Accuracy: *Accuracy* refers to closeness of agreement between a measured quantity value and a true quantity value of a measurand (VIM, 2.13). If one considers only the numbers, then one might examine the quantitative equivalent considerations. These are *trueness* and *precision*.
- Trueness: *Trueness* is the closeness of agreement between the mean value obtained from a large series of test results and an accepted reference value; in the diagram above, trueness is represented by the black dot.
- Precision: *Precision* is the closeness of agreement between measured quantity values obtained by replicate measurements under the same or similar specified conditions (VIM, 2.15) – i.e., how dispersed the group of white dots are from each other and from the black dot (dispersion). Precision measures random contributions to uncertainty.

  Note: It is important to understand that trueness and precision are independent of each other. One has nothing to do with the other.

Bias: Bias is the difference between the expectation of a measurement result and an accepted reference value. For example, if we determined from a number of measurements that the average trueness of several results had a certain relationship to a reference value (for example were 10% higher), then we might expect further measurements made in the same way to be similarly biased. While trueness is a property of a result or set of results, bias is a property of a measurement method. Bias is normally determined by measuring trueness. In the diagram, if we planned to do more measurements, we would expect most results to be high and to the right (i.e., we expect the result not to be true due to bias).

3.0 NEED FOR TRACEABILITY

Analytical data are often used for comparative purposes such as conformity to a "standard" or "limit". To compare results from different laboratories or from the same laboratory at different times with known confidence, all labs must be using the same "reference points". These common reference points are achieved by establishing traceability; that is, calibrations that can be related to primary national or international standards (ideally S.I. units of measurement) through an unbroken chain of comparisons all having stated uncertainties.

Traceability and its Relation to Uncertainty

Analytical data are the result of measurements. No measurement, no matter how carefully made, can be completely free of uncertainty. One can think of a test result as a "best estimate" of a property and the uncertainty as how sure we can be of that estimate. Uncertainty does not imply doubt about the validity of the measurement; it quantifies confidence and therefore adds value to the test result. Part of the quantifiable "true" uncertainty is the uncertainty inherent in the measurement data, so the ability to evaluate and control these uncertainties is crucially important for appropriate data interpretation. Knowledge of measurement uncertainty allows the data user to draw valid conclusions with a known degree of confidence.

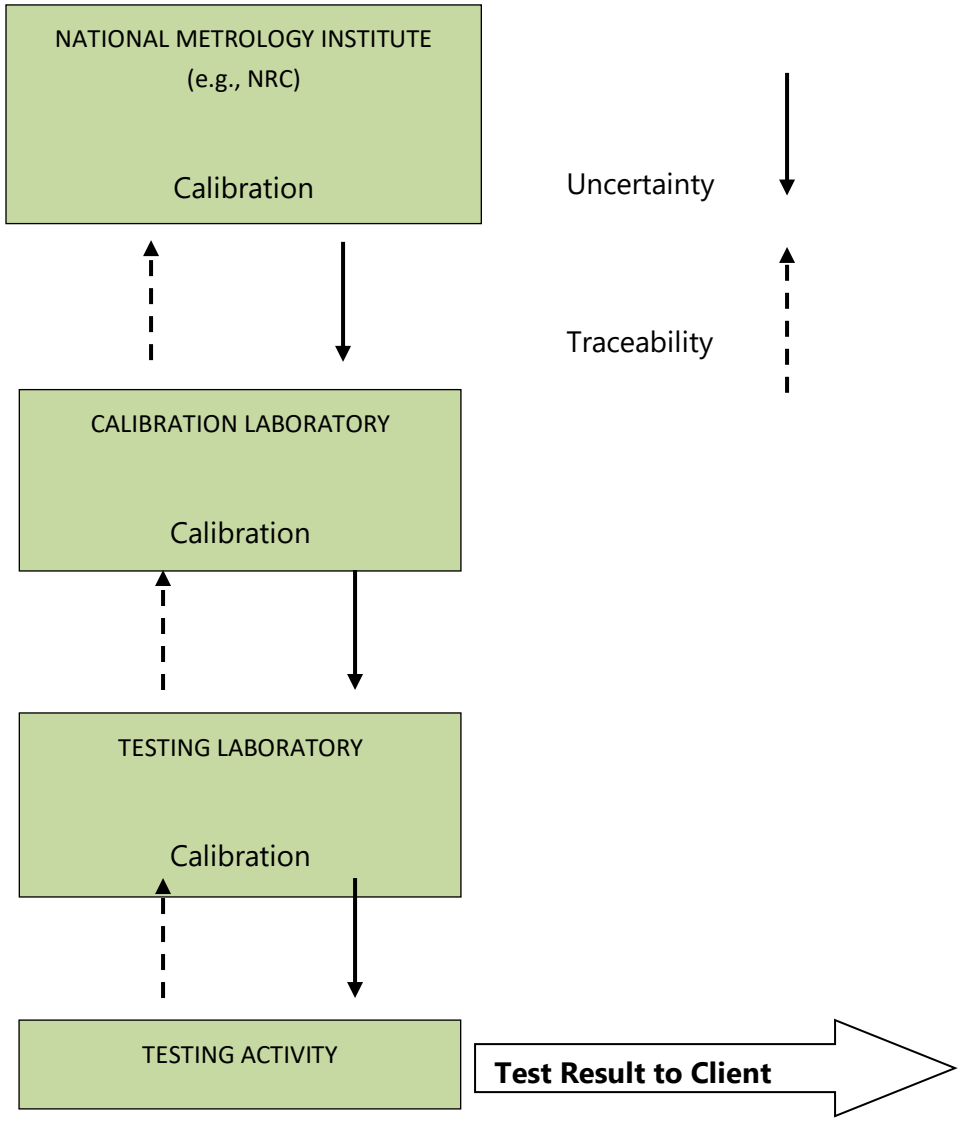
Agreement between labs (or within labs over time) is limited by the uncertainties in the traceability chain. So traceability places measurements on a consistent and comparable measurement scale. Uncertainty characterizes the strength of the links in the chain and characterizes the agreement to be expected between labs making similar measurements. In the traceability chain, uncertainty of test results is the uncertainty in the reference together with the uncertainty of making the measurement relative to the reference. While in a real chain the strength is determined by the weakest link, the strength of the traceability chain is determined by the collective strengths of all the links.

Traceability involves the competent propagation of uncertainties all along the chain of measurement to the test result produced by a testing laboratory. See the diagram on the following page. If uncertainties have not been propagated to the actual test result, the result is not traceable. If it is not traceable, it may appear precise but its trueness will always be suspect.

At the same time, uncertainties established along the traceability chain are the basis for the establishment of traceability of the measurement. The only property of a reference item (e.g., instrument, mass, reference material, etc…) that counts, in considering traceability, is uncertainty. Test results, in order to be traceable, must be conducted using traceable reference items (e.g., instruments, masses, reference materials, etc…) only.

Only those reference items that have been competently compared to others of known uncertainty (calibrated) can have their contribution to the overall uncertainty of the measurement objectively examined. When this condition is met, the measurement is considered traceable. If the reference items are not traceable, then the test result is not traceable.

3.1 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 NMI's 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 characterise a parameter, such as *mass* or *temperature*, to a specific level of uncertainty. They propagate this uncertainty in support of competent measurements. This affects legal measurements (butcher weigh scales and gas pumps) as well as other fields of science requiring competent measurement.

Each NMI has the ability to conduct measurements with very small uncertainties. They are also able to calibrate the instruments from calibration laboratories seeking to establish traceability to the NMI. This is the start of the traceability chain for a testing laboratory.

Calibration, *Traceability* and *Uncertainty* are all required at any point in the traceability chain. None of these are deemed to be present unless ALL are present.

4.0 IMPLEMENTATION OF A61-01 CALA *TRACEABILITY POLICY*

The CALA *Traceability Policy* (A61-01) means three things to CALA laboratories. First, it requires CALA laboratories to make use of measurement instruments, whose measurement traceability goes all the way back to an international standard (the SI) through a national measurement laboratory (e.g., NIST or NRC).

Second, it means that CALA laboratories must know how to spot the signs that a measurement result is traceable or not.

Third, it means that CALA 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.

NO CALIBRATION

= NO UNCERTAINTY

= NO TRACEABILITY

4.1 Selecting a Calibration Laboratory

4.1.1 General

Traceability means that all of the comparisons back along the Calibration chain to the national measurement laboratory were done by competent organizations (calibration laboratories). The competence of a calibration laboratory is most easily established by ensuring that the calibration laboratory is *accredited* to ISO/IEC 17025 for the task. For example, the following table represents a sample Scope of Accreditation for an accredited calibration laboratory:

Measurement Parameter	**Range of Measurement**	**Related Uncertainty**
Temperature	0.01 - 140.01 degrees C	+/- 0.003 degrees C
Volume	0.05 - 200.00 Litres	+/- 0.005 ml

An accredited calibration laboratory that is recognized by the CALA may be found on the websites of recognized accreditation bodies. E.g.

- Standards Council of Canada (SCC) – www.scc.ca.
- National Voluntary Laboratory Accreditation Programme (NVLAP) – www.nist.gov/nvlap
- A2LA – www.a2la.org
- ANSI-ASQ National Accreditation Board (ANAB) – www.anab.org

This list is not exhaustive as CALA recognizes other laboratory accreditation bodies.

4.1.2 Calibration Certificates

The following information should appear on the calibration certificate:

- An indication that the calibration laboratory is accredited to ISO/IEC 17025 by a recognized and named accreditation body. Remember, a service provider that is only registered (or certified) to ISO 9001 (or other management system standard) is not considered a technically competent calibration service provider.
- The serial number of the measuring equipment (or reference standards) being used to calibrate your equipment and a statement how the measurements are metrologically traceable to SI units; and,
- The measurement range for which your equipment was calibrated and the specific uncertainty measurements for that range.

Before purchasing a traceable thermometer or calibration certificate, you may be able to obtain from your potential supplier an example of their calibration certificates. This will allow you to review the calibration certificate and if you have any concerns about whether you are meeting the traceability and calibration requirements, you can get answers from your supplier before you purchase the item or service.

If newly purchased equipment is not provided with an appropriate calibration certificate, then a calibration must be performed prior to use.

4.2 Determination of Calibration Intervals

As a general rule, calibration at regular intervals cannot be eliminated simply because over time, there is 'wear and tear' on equipment and the measurement uncertainty of the test result is likely increasing. However, calibration intervals are not static, and understanding and applying a 'significance test' can assist the laboratory in determining logical calibration frequencies. Significance can be established using an approach that compares results from a simple statistical expression.

Any number, *x*, that is one third or less than another, *y*, will tend to have no significant effect on the outcome of the square root of the sum of their squares:

$$z = \sqrt{x^2 + y^2}$$

If, x = 1.0 and y = 3.0, then z = 3.16 if x is included in the expression and 3.0 if it is not. The difference is approximately 5%, a relatively insignificant difference when considering uncertainties.

Any number, *x*, that is one tenth or less than another, *y*, will tend to have negligible effect on the outcome of the square root of the sum of their squares.

If, $x = 1.0$ and $y = 10.0$, then $z = 10.05$ if x is included in the expression and 10.0 if it is not. The difference is approximately 0.5%, a *negligible* difference when considering uncertainties.

The following three examples may be used by laboratories to determine which of their instruments meet the requirement for significant effect in their calibration program.

- Example 1: If the uncertainty of an instrument is smaller than one-tenth of the largest contributor in the estimation of uncertainty for all tests affected by that instrument, then the laboratory has evidence to increase the period of calibration from one to two years or more, decreasing its frequency of calibration.
- Example 2: If the uncertainty of an instrument is between one tenth and one-third of the largest contributor in the estimation of uncertainty for all tests affected by that instrument, then the laboratory should maintain the period of calibration at one year.
- Example 3: If the uncertainty of an instrument is larger than one-third of the largest contributor in the estimation of uncertainty for all tests affected by that instrument, then the laboratory has evidence to decrease the period of calibration to less than one year; for example, every six months.

The choices to a laboratory are the following, and they depend on the significance of the contribution of the instrument to the overall uncertainty of the desired test result as described by the examples above:

- When instrument performance is very much better than the requirement, the laboratory can reduce the calibration frequency (extend the cycle time). See example 1 above.;
- When performance is at the same level as requirement, the calibration frequency must be increased. See example 3 above; or,
- When the laboratory does not know, best practice is to calibrate that equipment once per year. See example 2 above.

In general, the only instruments in a typical analytical laboratory that require traceability through external calibration are those related to mass and temperature and subsidiary measurements (e.g., barometric pressure). There may be some circumstances where traceable volume measurements are required, but in this instance, traceability is usually linked to mass.

4.3 Balances

4.3.1 Calibration

Daily use of a balance often leads to subtle changes in the balance. This may be seen when reviewing the verification data and may also be indicated when the "as found" checks on the balance by the calibration laboratory are found to be out-of-specification compared to acceptance

criteria and the "as-left" conditions. If these are all found to be in-specification, then perhaps the balance is stable enough to reduce the calibration frequency IF the measurement uncertainty of the balance at the lab is significantly less than the criteria required for the weighing (for example, if MU= 0.001 mg but the balance is only used to measure down to 0.1 mg). In these cases, the frequency might be decreased but not eliminated. Keep in mind, too, that other valuable functions may be performed at the same time as the calibration (e.g., inspection of the balance, cleaning, checking of the verification weights, etc.).

4.3.2 Verification

Verification allows the laboratory to demonstrate stability of operation of the balance over the whole range normally used. The criteria for these checks should be fit-for-purpose.

The documented procedure for the verification process should include:

- the frequency of verifications;
- the use, storage, and handling of appropriate weights;
- acceptance criteria; and,
- actions to be taken when the acceptance criteria are not met (e.g., recall of results, etc…).

4.4 Thermometers and Thermocouples

4.4.1 Calibration

The frequency of recalibration of thermometers will be dependent on the use of the thermometer. For example, incubator thermometers with a narrow temperature requirements should be calibrated more frequently than those used for sample storage. Liquid-in-glass thermometers being ramped up and down will likely require more frequent calibration than a liquid-in-glass thermometer maintained at one temperature, as will thermocouples and thermistors.

A laboratory may demonstrate traceability of their temperature measurements through external calibration using an accredited calibration laboratory, or performing internal calibrations.

A laboratory may opt to send working thermometers to an accredited laboratory for calibration at an established frequency (e.g., annually). This is by far the easiest way to ensure traceability of all critical thermometers but can be costly and requires the purchase of more thermometers than are used on a daily basis in order to ensure the availability of calibrated thermometers when others are being calibrated.

A laboratory may decide to maintain a limited number of thermometers that are sent to an accredited calibration laboratory on a scheduled basis. The laboratory may then use these reference thermometers to calibrate working thermometers.

Thermometer and thermocouple calibrations and verifications against the laboratory's reference thermometer are typically conducted at a single temperature (e.g., temperature of use or ice-point).

4.4.2 Verification

Thermometers exposed to wide fluctuations in temperature may require frequent verification. Similarly, any temperature device being moved frequently may also require a higher frequency of verification (e.g., a working thermometer being moved from incubator to incubator).

4.5 Volumetric Measurements

4.5.1 Calibration

Dispensing devices must be calibrated upon receipt and at an established frequency (e.g., annually) and verified.

Dispensing devices may be sent to an accredited calibration laboratory for calibration. Alternately, the laboratory may perform an internal calibration following a generally accepted procedure. This procedure generally involves 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. Acceptable performance (precision) is based on the precision required for the piece of dispensing equipment. Historical daily or as-used verification data can be used to develop standard deviations for calibration provided all the requirements for calibration are met for each verification reading. This includes, but is not limited to, ensuring:

- that the laboratory documents their verification procedure to include all of the same requirements for the performance of a calibration but with as few as one reading on each day of verification;
- that a statistically valid number of readings are used in the determination of the standard deviations for the calibration;
- that the person conducting the verification has been properly trained on the procedure used;
- that they can competently propagate uncertainties from the reference standard (device) to the working instrument; and
- that they use a calibrated balance that meets the requirements of ISO/IEC 17025 for this purpose.

4.5.2 Verification

Daily or as-used verifications are performed by dispensing a measured volume to a tared balance and recording the weight (corrected for temperature and pressure). For adjustable dispensing devices, this procedure should be performed at more than one volume.

While syringes generally come with certificates from an ISO 9000 registered company, this certificate in itself is not sufficient evidence of on-going integrity of the syringe. Syringes used for standard preparation and sample introduction for the GC etc. can be verified indirectly through the analysis of surrogates or internal standards.

5.0 RECOMMENDED CALIBRATION AND VERIFICATION INTERVALS

The following table sets out recommended calibration and verification intervals.

Equipment	Calibration Interval	Verification Frequency
Balances	Initial and every two (2) years	Daily or as-used
Masses • Reference • Working	Initial and every five (5) years	Day that balance is calibrated by an external calibration provider. Day that balance is calibrated by an external calibration provider.
Thermometers • Reference • Working	Initial and every five (5) years Initial and every two (2) years	Annually, using ice-point or boiling point (liquid-in-glass only). Annually, against the reference thermometer.
Pipettors	Initial and annually	Daily or as-used

6.0 TERMS AND DEFINITIONS

- Calibration: Calibration is a comparison of measurements between two standards or measurement devices. It involves the competent propagation of uncertainties from the instrument or standard whose measurement characteristics are known and traceable to the SI, to an instrument or standard whose measurement characteristics are to be quantified through this comparison.
- Calibration Curve: This applies to analytical laboratory instrumentation. The term defines the relation between analyte concentration and instrument response.
- Control Standard: A standard used as a basis for comparison with calibration standards, prepared independently from the calibration standards, and which undergoes sample processing identical to that carried out for the calibration standards.
- Reference Material (RM): Material sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process (ISO 17034:2016)
- SI (Système International d'Unités): The name *(International System of Units)* adopted by the 11th General Conference on Weights and Measures (1960) for the recommended practical system of units of measurement. The **base units** are a choice of seven well-defined units: the metre, the kilogram, the second, the ampere, the Kelvin, the mole, and the candela.
- Traceability: A property of the result of a measurement or the value of a standard whereby it can be related, with a stated uncertainty, to stated references, usually national or international standards, through an unbroken chain of comparisons. (ISO Guide 30).
- Trueness: The closeness of agreement between the average value obtained from a large series of test results and an accepted reference value. (ISO 3534-1, 3.12).
- Uncertainty of Measurement: Parameter that characterizes the dispersion of the quantity values that are being attributed to a measurand, based on the information used. (VIM)
- Verification: Confirmation through examination of a given item and provision of objective evidence that it fulfils specified requirements. [modified from ISO 9000:2015, item 3.8.12]