Sections in This Document

1. Purpose

To provide guidelines for the establishment and maintenance of metrological traceability of measurement results by means of a documented unbroken chain of calibrations, each contributing to the measurement uncertainty, linking them to an appropriate reference.

2. Scope

This procedure describes how the Office of Regulatory Science (ORS) laboratories achieve measurement and/or metrological traceability for their measuring and testing equipment, as well as for its reference standards, materials, and media.

3. Responsibility

ORA laboratories are responsible for maintaining records of measurement and metrological traceability. These records include, but are not limited to, the calibration and verification of equipment such as balances, pipettes, and thermometers. Records also must include the certificates for reference standards and materials used to calibrate laboratory equipment and for reference cultures.

4. Background

None

5. References

A. ISO/IEC 17025:2017, General requirements for the competence of testing and calibration laboratories, Section 6.5 & Annex A (Informative): Metrological Traceability.

B. ISO 17034, General requirements for the competence of reference material producers.

C. AOAC International Guidelines for Laboratories Performing Microbiological and Chemical Analysis of Food, Dietary Supplements, and Pharmaceuticals – An Aid to Interpretation of ISO/IEC 17025:2017; August 2018.

D. EURACHEM/CITAC guide Traceability in Chemical Measurement – A guide to achieving comparable results in chemical measurement

E. ILAC Policy on Traceability of Measurement Results

6. Procedure

6.1. Measurement Traceability

A. Reference balance mass units, thermometers and thermocouples are traceable to International Standard (SI) units of measurement.

Calibration laboratories providing these reference standards that conform to and are accredited to ISO 17025:2017 provide metrological traceability with their certificates.

B. Laboratory measuring equipment is calibrated or standardized using materials of known and acceptable purity (i.e. neat compounds, or

reference materials of know composition traceable to national, international or equivalent standards). Uncertainty contributions are addressed in ORA-LAB.5.4.6. Estimation of Uncertainty of Measurement.

Certified values of certified reference materials from reference material producers conforming to ISO 17304 provide metrological traceability.

C. Media are verified against national or international standards (i.e. reference cultures or certified reference cultures).

D. The traceability to SI units is achieved by reference to national measurement standards that are primary realizations of the SI units or agreed representations of SI units. SI units are based on fundamental physical constants of mass, distance, and time.

E. Traceability may also be established through secondary standards that are calibrated by a national metrology institute such as the National Institute for Standards and Technology (NIST).

6.2. Traceability Alternatives to SI Units

A. Where calibrations cannot provide traceability to SI units, confidence in measurements is established with traceability to measurement standards such as:

1. certified reference materials provided by a competent supplier to give a reliable physical or chemical characterization of a material, and
2. the use of specified methods and consensus standards that are clearly described and agreed to by the parties concerned.

B. Where traceability of measurements to SI units is not possible or not needed, the same requirements for traceability, for example, certified reference materials, agreed methods or consensus standards are needed.

C. For some analyses certified reference materials are not readily found. In this case, a material with similar properties and stability is selected. The properties of interest in this material are characterized by repeat testing, preferably by more than one laboratory and using a variety of methods.

D. Where national or international standards are not found for the verification of the performance of microbiology procedures, the laboratory records recovery by either participating in a program of interlaboratory comparison or proficiency testing, use of reference

cultures or certified reference cultures, or by a mutual agreement with client on expectations.

E. In cases where a Reference material is made in-house the laboratory should comply with ISO 17034 General Requirements for the competence of reference material producers.

6.3. Calibration and Calibration Services for Reference Standards

A. Laboratory reference standards (i.e. weights, thermometers, etc.) are calibrated by an accredited calibration laboratory that can provide the laboratory with certificates of calibration linking the calibrations to measurements made by NIST or some equivalent. The identity of the calibration laboratory is included on the certificate as well as the methods and standards primary to the process.

B. Traceable reference thermometers and thermocouples are calibrated every two years, and reference mass units are calibrated every five years. Frequency of other reference standards is documented in laboratory procedures.

C. Reference standards are used for calibration verification only and not as working standards.

D. A calibration certificate bearing an accreditation body logo from a calibration laboratory accredited to ISO/IEC 17025 for the calibration concerned is sufficient evidence of traceability of the calibration data reported. NOTE: the calibration laboratories must be accredited to ISO/IEC 17025:2017 after August 2020.

E. The date of calibration is included for the reference standard in the calibration certificates.

F. These reference standards are stored in a protected area to prevent any damage that would invalidate their use.

G. Servicing is requested from an accredited calibration laboratory when data acceptance criteria for verification are not met and the analyst is unable to implement a corrective action.

6.4. Reference Materials

A. Reference materials are to be traceable to SI units of measurement, to standard reference materials (SRMs), certified reference materials (CRMs), certified reference cultures (CRCs) and obtained from reference material providers accredited to ISO 17034 when possible.

B. In microbiology, the organisms required for the tests shall be verified, stored appropriately, checked for purity and demonstration of

biochemical or other biological characteristics, as appropriate for their application.

C. The organisms are traceable and documented from date of possession.

D. Manufacturer's Certificates of Analysis are retained for reference materials used.

E. A listing of reference materials in use in the laboratory is maintained. This listing contains, as a minimum, the chemical name or description, source, manufacturer's lot number, unique laboratory identification number (if lot number not used), date of receipt, manufacturer's expiration date if available or laboratory determined expiration date, open date when open date impacts expiration date, and traceability to person assuming responsibility for the reference material.

F. Reference materials are labeled using an identification scheme that allows the laboratory to trace the lot used in any analysis, date of receipt and initials, date opened and initials, and expiration date.

G. Analytical records include the identity of and the measurement values for reference materials used in the routine analyses. This information provides traceability to the measurements for batches of samples measured sequentially to the reference material. This information is recorded in the analytical worksheets, instrument logbooks or incorporated in the instrument print out for the analyses.

H. Calibration of instruments is verified during the performance of analyses. The frequency of calibration is identified in the test method or per laboratory instrument procedure.

I. The safe handling, transport, storage, and use of laboratory reference materials are conducted according to the manufacturer's instructions in order to prevent contamination, deterioration, and to protect their integrity.

J. Reference materials are not used past their expiry date without documented requalification demonstrating they are still suitable for use.

K. Reference materials with an expiration date of only month and year will expire on the last day of the month.

L. Reference materials are not stored with samples.

6.5. Reagents, solvents, gases, and media

A. For most analyses, analytical reagent grade is satisfactory. Trace analyses frequently call for special ultra pure reagents, solvent and gases.

B. Reagent grade chemicals should be ordered in quantities such that the supply will be consumed within the manufacturer's expiration date or 5 years, whichever is first.

C. Chemicals and solvents with an expiration date of only month and year will expire on the last day of the month.

D. For extended use of purchased laboratory reagents and solutions without a "use by" or expiry date provided by the manufacturer, an assessment will be conducted (literature review may be acceptable) of that specific chemical's or chemical family's stability and a "use by" or expiry date be determined. The determined "use by" or expiry dates are documented.

E. All reagents, solutions and media are labeled with date of receipt and initials, date opened or prepared and initials, and expiration date.

F. Reagents, solvents, gases, and media are stored according to the manufacturer's directions and are not kept longer than recommended by the manufacturer or the method. Reagents are checked for signs of deterioration. Reagents that have deteriorated are not used and are discarded in accordance with a Laboratory's Safety and Hazardous Waste procedures.

G. Media purchased or prepared are evaluated for suitability.

6.6. Disposal

Reference materials, reagents, solvents, gases, and media are disposed of according to federal, state and local regulations. The laboratory has procedures for the proper disposal.

7. Glossary/Definitions

A. Calibration – A calibration is the set of operations which establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system, or values represented by a material measure, and the corresponding known values of known values of a measurand.

B. Calibration laboratory - a calibration laboratory provides calibration services as its principal activity. This type of laboratory is also referenced as a metrology laboratory.

C. Certificate of Analysis – a document issued by the Quality Assurance section of a manufacturer that confirms a product meets its product specification. They commonly contain the actual results obtained from

testing performed as part of quality control of an individual batch or product and include Measurement Uncertainty information.

D. Certified reference cultures (CRC) – These are cultures traceable to nationally or internationally recognized type culture collection (i.e. American Type Culture Collection).

E. Certified reference material (CRM) – A material with one or more property values certified by a technical valid procedure, accompanied by or traceable to a certificate or other documentation that is issued by a certifying body.

F. International standard – These are standards from an international repository that fulfill the properties of primary standards for the realization of SI units.

G. ISO 17034 - specifies general requirements in accordance with which a reference material producer has to demonstrate that it operates, if it is to be recognized as competent to carry out the production of reference materials.

H. Logbook – Record containing specified information. There is no requirement that the logbook be a bound notebook or in a binder. Records may be kept electronically.

I. Measurand – A measurand is a particular quantity subject to measurement.

J. Measuring and testing equipment – These are devices used by the laboratory for testing and measurement. Equipment, instruments, and instrumentation are terms used synonymously with measuring and testing equipment.

K. National Standard – These standards are from a national repository that fulfills the properties of primary standards for the realization of SI units.

L. NIST – National Institute of Standards and Technology – Nationally recognized measurement body for the United States.

M. Reference Material (RM) – a material with one or more property values sufficiently well established to be used for calibration (standardization) of an apparatus, the assessment of a measurement method, or for assigned values to materials; sometimes referred to as Standard Reference Material (SRM).

N. Reference Standard – a standard with generally the highest metrological quality available at a given location in an organization, from which measurements made there are derived. Generally, this refers to

recognized national or international traceable standards such as NIST thermometers and weights.

O. Standardization – This is the assignment of a compositional value to one standard (measurand) based upon another standard.

P. Traceability – Traceability is an unbroken chain of calibrations or comparisons to identified primary standards of the SI units of measurement.

Q. Verification – Verification is confirmation by examination and provision of evidence that specified requirements have been met.

8. Records

A. Certificates of calibration for Reference standards

B. Certificates of analysis for Reference materials

C. Inventory of Reference Materials and Reference Cultures

D. Reference Material logbooks

E. Reference material expiration extensions

F. Assessments and/or literature used for assessments for laboratory determined expiry dates

9. Supporting Documents

A. ORA Laboratory Manual, Volume II

B. ORA Laboratory Manual, Volume II, ORA-LAB.5.5 Equipment

C. Laboratory Hazardous Waste Management Plan

D. Laboratory Chemical Management Plan

E. Microbiology Laboratory media procedure for preparation, labeling and quality control

10. Document History

Revision #	Status* (D, I, R)	Date	Author Name and Title	Approving Official Name and Title
1.2	R	11/16/05	LMEB	LMEB

1.3	R	12/06/06	LMEB	LMEB
02	R	05/15/2019	LMEB	LMEB

* - D: Draft, I: Initial, R: Revision

11. Change History

Revision #	Change
02	Revisions made to align this procedure with new ISO/IEC 17025 and AOAC requirements. Revision to formatting and policy clarifications were also made.

12. Attachments

None