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ORNL Guidelines for User Calibration -- SPRUCE Implementation

Introduction

This document is the SPRUCE adaptation of the "ORNL Guidelines for User Calibration" [https://sbms.ornl.gov/sbms/sbmsearch/subjarea/CALIBRATION/UserCAL.htm]. Our intent is to implement these guidelines in our SPRUCE Experiment context and use them to make informed decisions regarding calibration of SPRUCE equipment.

ORNL Measuring Equipment Calibration Guidelines

Calibration routines ensure measuring equipment is periodically checked and adjusted to maintain accuracy within necessary limits. A graded approach to implementing calibration procedures is encouraged.

- Measuring equipment essential to safety-systems, environmental-permitted systems, security
 system, or other mission essential operations are subject to formalized calibration routines
 performed by certified or qualified personnel. It is suggested that a <u>recall program</u> under one
 of the ORNL calibration groups be used whenever possible if formalized routines are needed.
 - Recall Program: A database or other program which automatically monitors the calibration status of a piece of measuring and/or test equipment and notifies the appropriate responsible person when the device has exceeded its calibration time interval.
- Equipment used for research or operations that fall outside of the applications above may be considered for user-calibration commensurate in formality with the application and/or as recommended by the manufacturer.

SPRUCE Implementation of Calibration Guidelines Using a Graded Approach

SPRUCE investigators will engage services of ORNL Calibration Groups or Calibration Service Vendors for <u>selected</u> equipment and implement user calibration routines as applicable for research equipment. Measuring equipment used by the SPRUCE experiment staff generally meets the guidelines for implementing user-calibration routines.

Calibration decisions (calibration group or user calibration) will be made based on a number of factors:

- manufacturers recommendation for calibration
- cost of calibration vs. replacement of a sensor
- calibration interval and spare instrument stability during storage

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- feasibility of successful calibration or verification of calibration given an instruments physical location (e.g., on a tower with some "flexibility")
- redundancy of specific instruments/measurements that allow detection of instrument drift, instability, and failure
- availability of calibration vendors for specific equipment

Calibration decisions for measuring equipment will be documented.

Calibration Overview

- Measuring equipment will be identified and records maintained of who can calibrate the measuring equipment to meet the applicable calibration requirements.
- Calibrations should be performed by certified calibration organizations or technically capable
 users in accordance with appropriate calibration methods, such as quality plans, operating
 procedures, analytical methods, and recommendations from manufacturers. In research, the
 calibration method is typically documented as part of the research to be included with reports
 on the results.
- Calibration procedures (e.g., ICS/CSEH1010 Rev. 03, for Humidity/Temperature Sensor) and proven user calibration routines will be implemented.
- Instruments, sensors, and equipment will be successfully calibrated per applicable methods prior to or at the time of long-term installation in the field.

Calibration Guidelines Applicability

- ORNL SPRUCE investigators will follow these guidelines for the calibration of field instruments and sensors installed at the experimental site in Minnesota.
- ORNL SPRUCE investigators performing laboratory analyses will follow their standard calibration procedures.
- SPRUCE Collaborators, with funding from DOE or other sources, will be responsible for their calibration routines for field and laboratory instruments.

SPRUCE User Calibration Implementation

Identifying Measuring Equipment

- SPRUCE Equipment is labeled with bar codes and entered into the ORNL Facility Service Center (FSC), Asset Management application along with the equipment characteristics and indexed to the SPRUCE project and specific installation location.
- For measuring equipment with multiple users (e.g., LiCor 6400s), a process will be implemented to designate calibration status. Examples include placing a calibration sticker

- on the equipment, adding a notation in an instrument log, or keeping a list of calibrated equipment. Calibration status need not be designated for items for which calibration or calibration verification is performed before use.
- Equipment listed in the Facility Service Center, Asset Management, will be entered in the Calibration Recall Program per SPRUCE calibration decisions.
- Any SPRUCE Equipment not entered into FSC, will be recorded and tracked by the Instrument Custodian.

Calibration and Calibration Verification Procedures, Routines, and Methods

- Calibration procedures, routines, and methods will be identified for measuring equipment.
- Existing procedures of ORNL calibration groups will be utilized whenever possible (e.g., ICS/CACZ2000, CALIBRATION OF LI-COR BIOSCIENCES Model LI-840A CO2/H2O Gas Analyzer and ORNL Metrology, VAISALA HMP155 TEMPERATURE VERIFICATION, Revision: 1.0, Compiled 2014-01-31).
- If necessary, SPRUCE-specific procedures and routines will be developed.
- Newly developed procedures may follow the Instrumentation and Control Service (ICS) format and be controlled and distributed per ICS policies.
- SPRUCE-specific routines may incorporate the equipment manufacture's recommended procedure for and frequency of calibration and calibration verifications.

The following attributes should be defined for measuring equipment and included in calibration procedures:

Frequency

- Calibrations should be performed or verified at appropriate intervals, before use, as needed, or whenever accuracy is suspect. For some measuring equipment, such as analytical instrumentation, it is generally advisable to verify the accuracy of a calibration before, during, and after the measuring activity using calibration check standards independent of the standards used for calibration (i.e., different lot number or different vendor).
- Frequency of user calibration of equipment will be established and documented in calibration routines.
- Frequency of calibration may be entered in the Facility Service Center, Asset Management, Calibration Recall Program per SPRUCE calibration decisions.
- Calibration frequency may be adjusted after a couple calibration cycles to either a shorter or longer interval depending on the stability of the calibration. The recall cycle can be adjusted as needed.

Accuracy

• The level of accuracy for a calibration should match or exceed that needed for the measurements. In research, accuracy must be at least that required to meet professional

standards enforced by the peer review process. Measurements supporting operations need a level of accuracy able to meet sponsor/customer expectations or other applicable requirements.

• The level of accuracy is reflected by the acceptance limits defined for the calibration.

Calibration Standards and Characterization

- Calibrations should be performed using suitable standards. Wherever applicable, calibration standards should be traceable to a nationally or internationally recognized source, if available. If a recognized source is not available, materials used for standards should be characterized. Characterization may be achieved by repeated analysis, preferably by two different techniques. In some cases, vendor specifications may provide acceptable characterization information.
- Chemical or gas standards may have an expiration date that needs to be monitored and the standard replaced or re-characterized when that date is reached.
- Equipment that may be user calibrated before use (e.g., balances), may rely on certified standard weights that require periodic re-certification.
- Documentation certifying or characterizing materials used as standards should be handled in a manner appropriate for the work performed. This may include maintaining documentation in a way that enables users to trace a calibration from the equipment calibrated to the standard(s) used for the calibration to the documentation certifying or characterizing the standard(s). No documentation is needed for equipment for which pre-use calibration is an automated and fail-safe part of their use.

Training

Calibrations should be performed or verified by technically capable users. Documentation of
users with the technical ability and administrative responsibility for calibrating equipment
will be maintained.

Out-of-calibration Condition

- User Calibration
 - When equipment is found (AS-FOUND) to be out of calibration or calibration cannot be verified, the <u>user</u> should take appropriate action to prevent the inadvertent use of the equipment, document the problem, and determine the impact on previously acquired measurements.
 - o Notify the <u>Instrument Custodian</u> who will prevent the inadvertent use of measuring equipment that cannot be calibrated or calibration verified, equipment should be

designated as out of service or managed in a way that keeps it from being used for measurements.

- o Notify the **<u>Data Manager</u>** when calibration problems will impact collected data.
 - Depending on the measurement, one of the following data management actions may then be taken:
 - setting affected results to missing or flagging them as suspect,
 - using algorithms to gap-fill affected results and flag them as calculated values,
 - repeating measurements after the equipment is calibrated, or
 - additional measurement-specific actions as may be needed.

• ORNL Calibration Groups and Calibration Service Vendors

- When equipment is found (AS-FOUND) to be out of calibration or calibration cannot be verified, the *calibration organization* should take appropriate action to prevent the inadvertent use of the equipment.
- Notify the <u>Instrument Custodian</u> who will prevent the inadvertent use of measuring equipment that cannot be calibrated or calibration verified, equipment should be designated as out of service or managed in a way that keeps it from being used for measurements.
- o Instrument Custodian will determine the impact on previously acquired measurements.
- o Instrument Custodian will notify the **<u>Data Manager</u>** when calibration problems will impact collected data.
 - Depending on the measurement, one of the following data management actions may then be taken:
 - setting affected results to missing or flagging them as suspect,
 - using algorithms to gap-fill affected results and flag them as calculated values,
 - repeating measurements after the equipment is calibrated, or
 - additional measurement-specific actions as may be needed.

Documentation and Communication of Calibration Results

- For equipment listed in the Facility Service Center FSC), Asset Management, documentation of the results of calibration, as defined in the respective procedure (e.g., AS-FOUND, AS-LEFT, and out-of-calibration problems), will be attached (digitally) to that equipment's record.
- For equipment listed in the Facility Service Center, Asset Management, communication of results may be via e-mail to the Instrument Custodian and Data Manager.
- For non-FSC equipment, documentation of calibration per the procedure may be in the Researcher's Field or Laboratory Notebook.
- For non-FSC equipment, communication of results to the Instrument Custodian, Team members, and Data Manager is the responsibility of the Researcher.

• For equipment calibrated by ORNL Calibration Groups and Calibration Service Vendors the documentation of results will be specified in the work agreement and those results will be communicated to the Instrument Custodian.

Documentation and Communication of Calibration Verification Results

- Calibration verification (i.e., checks) will be specified for measuring equipment, as applicable, in procedures. Calibration verification is typically performed before and after use.
- Documentation of calibration verification per the procedure may be in the Researcher's Field or Laboratory Notebook or recorded on a Data Collection Form.
- Communication of results to the Instrument Custodian (if different from Researcher), Team members, and Data Manager is the responsibility of the Researcher. In particular, when the measuring equipment cannot be calibrated or calibration verified, notify the appropriate Team members for equipment service.

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Roles and Responsibilities for Implementation of SPRUCE Equipment Calibration

Role	Responsibilities	Notes
Project Manager	Approve Calibration Decisions	
SPRUCE Team Researchers, Modelers, Instrument Custodians, Technicians, Data Managers	 Define Equipment Calibration Needs Frequency/recall program Accuracy Standards Calibration steps Calibration verification steps Training Documentation 	
Instrument Custodians	 Implement Calibration Guidelines Identify measuring equipment Document calibration decisions Structure calibration and calibration verification requirements and steps into procedures, routines, and methods documents Implement calibration recall schedule Monitor calibration and calibration verification results For out-of-calibration equipment, take action to prevent further use and document problem 	Calibration decision: Who will perform calibration? SPRUCE user calibration or ORNL calibration organization, or external third-party calibration vendor?
Technically Capable Users	 Perform User Calibrations Apply calibration and calibration verification methods to applicable equipment 	Training may be required.
ORNL Calibration Groups and Calibration Service Vendors	Perform CalibrationsApply calibration procedures to applicable equipment	
Data Managers	 Connect Measuring Equipment with Collected Data For out-of-calibration equipment, identify any collected data and take action to prevent inadvertent use Substantiate problem and adjust or qualify data as needed 	