

SOP 49
Standard Operating Procedure
for
Calibration of Environmental Monitoring Standards by Direct Comparison

1 Introduction

1.1 Purpose

The purpose of this procedure is to use calibrated environmental sensors/instruments as short-term transfer standards immediately after its calibration to perform downstream calibration of internal laboratory environmental sensors/instruments for calibration, verification that measurement results continue to be stable, or for validation of extending calibration intervals. The procedure may be suitable for sensors/instruments that are used for laboratory monitoring where a larger uncertainty may be acceptable and possibly for other calibration uses provided the application and required uncertainties are evaluated and found to be acceptable.

1.2 Range and Scope

Detailed measurement ranges, standards, equipment, and uncertainties for this SOP are generally compiled in a separate document in the laboratory (e.g., traceability hierarchy, uncertainty tables). Uncertainties of this process must be assessed (Section 5.5) to determine if they are sufficiently small for the standards to be used in downstream calibrations or if uncertainties are only suitable for laboratory monitoring applications.

1.3 Limitations

The temperature range is limited from 15 °C to 25 °C if based on typical laboratory conditions; the range in air may be from 5 °C to 95 °C if a wider range of calibrated standards and whether an oven, temperature bath, or ability to vary conditions are available/possible. Barometers are compared under various laboratory ambient pressures. Relative humidity is compared over during several days with various ambient laboratory humidity conditions unless a humidity chamber is available or the laboratory can vary conditions.

If multiple points are obtained during varying conditions observed in the comparisons, such as through use of a temperature bath/oven or use of relative humidity salt solutions, only the range of measurements between multiple calibration points may be considered suitable for calibration applications, provided the uncertainty is sufficiently small. If the prior calibration(s) of the instrument being calibrated demonstrated a best fit linear response where all offsets from nominal were within the stated uncertainty, a one-point calibration may be used. When a one-point calibration is used, such as at standard

environmental conditions only, the instrument should only be used for environmental monitoring and should also be verified again at one-fourth of the intended calibration interval to ensure ongoing accuracy and stability under laboratory environmental conditions.

1.4 Prerequisites

1.4.1 Staff must have suitable training and have demonstrated proficiency in comparison calibration measurements, e.g., those used in temperature, mass, volume, and dimensional parameters.

1.4.2 Valid calibration certificates that are less than 30 days old, with appropriate values and uncertainties, must be available for all the standards used in this calibration. All standards must have demonstrated metrological traceability to the international system of units (SI), which may be to the SI through a National Metrology Institute such as NIST. Exception: where the calibration of the standards is not less than 30 days old, enough laboratory data must be available to demonstrate historical stability of the instrument to the limits needed for this calibration.

1.4.3 Standards must be evaluated to ensure that standard uncertainties for the intended level of calibration are sufficiently small. The uncertainties resulting from this procedure may be suitable for monitoring laboratory conditions but not suitable for use in calibration processes unless the uncertainties of the standard calibration and the resolution of the standard are sufficiently small (i.e., the uncertainty of the instruments used as a standard must have better resolution than the instruments being calibrated if they are to be used as downstream measurements for calibrations).

1.4.4 Maintain environmental conditions within the limits noted in Table 1. These limits are not critical for this calibration; the limits are recommended based on good laboratory environmental control practices.

Table 1. Suggested Environmental limits for calibrations.

Temperature Range	Temperature Stability	Relative Humidity	Relative Humidity Stability
18 °C to 23 °C	± 1 °C	40 % to 60 %	± 2 %

2 Methodology

2.1 Summary

Laboratory environmental sensors/instruments are used as transfer standards to calibrate internal environmental sensors/instruments owned by the laboratory

using direct comparison procedures over a series of days. Results are used to provide for calibration, evaluation of the calibration status, or stability of measurement results to support calibration intervals.

2.2 Equipment / Apparatus

Limits given in this section are used to evaluate the final calibration results from this procedure when sensors/instruments will be used to provide corrections in subsequent calibrations (e.g., buoyancy corrections in mass or gravimetric volume calibrations; air density corrections used in dimensional calibrations). In this case, the calibrated uncertainty limits of the sensors/instruments to be used as standards must be significantly smaller than limits stated in this section (e.g., by enough so that conformity statements to these limits may be made based on measurement results and calculated uncertainties). See NISTIR 6969, SOP 2 for recommended limits for calculating air density.

When the following limits are not attainable, the final measurement results and uncertainties may be evaluated for suitability for use as laboratory monitoring sensors/instruments. Monitoring limits might consider 1 % of the air density limits as shown in SOP 2.

2.2.1 Calibrated barometer with sufficiently small resolution, stability, and uncertainty (See NISTIR 6969¹, SOP 2, e.g., accurate to ± 66.5 Pa (0.5 mm Hg)) to determine barometric pressure.¹

2.2.2 Calibrated thermometer with sufficiently small resolution, stability, and uncertainty (see SOP 2, e.g., accurate to ± 0.10 °C) to determine air temperature.¹

2.2.3 Calibrated hygrometer with sufficiently small resolution, stability, and uncertainty (see SOP 2, e.g., accurate to ± 10 percent) to determine relative humidity.¹

2.3 Procedure

2.3.1 Identify a sensor/instrument that was recently calibrated by an accredited supplier and where the calibration certificate has been evaluated to ensure suitable measurement results and uncertainties are available. Verify that the standard and any constants from calibration reports have been entered into their respective devices correctly (if applicable). This instrument will be used as the standard, *S*, for this comparison procedure.

¹ NISTIR 6969, Selected Laboratory and Measurement Practices, and Procedures to Support Basic Mass Calibrations. SOP 2, Applying Air Buoyancy Corrections.

2.3.2 Equilibrate the standard and the instrument to be calibrated, X , for 2 hours to 4 hours in a suitable laboratory environment. If the instruments have been outside of a laboratory environment, allow a 24 hour equilibration time.

2.3.2.1 Electronic equipment should be plugged in and warmed up as specified in their operating manuals.

2.3.2.2 Ensure that all barometers are at the same elevation and that station pressure values are used (not sea-level pressures).

2.3.2.3 All thermometers involved in the calibration should be placed in a stable medium (determined by sensor type). If a temperature oven or bath is available, it is preferred. However, a solid block or closed air system may be used for probes restricted to air; immersed block in water that is equilibrated to laboratory conditions may be used for liquid-compatible probes.

2.3.3 Option A. Obtain and record observed measurement values for the Standard, S , and Unknown, X , for temperature S_t , X_t , for pressure, S_P , X_P , and for relative humidity S_{RH} , X_{RH} , as shown on the Appendix A data sheet for Option A. Record observations over a period of three days, taking care to record or convert to similar measurement units. Record the measurements of each parameter within 30 s of each other to minimize potential drift due to laboratory environment or human influence factors in the measurement results. If additional degrees of freedom are needed, for example, where pooled standard deviations are not available, this process may be extended over more days. (Do not perform all three sets of observations on the same day).

2.3.4 Option B. If excess drift is suspected or observed in either the instruments or the environment, use two sets of observations in reverse order (S_1 , X_1 , X_2 , S_2) on each day (double substitution) for temperature, pressure, and relative humidity. Record data as shown on the Appendix A data sheet for Option B.

3 Calculations (Measurement Equations)

Perform the following calculations for each measurement parameter that is being calibrated (temperature, pressure, relative humidity) as applicable.

3.1 Calculate the three observed measurement differences, d_n , for each of the measurement parameters spread out over three different days:

3.1.1 To calculate the observed difference for Option A, use Eqn. 1 for temperature, pressure, and relative humidity observations (*obs*).

$$d_n = X_{obs} - S_{obs} \quad \text{Eqn. (1)}$$

3.1.2 To calculate the observed difference for Option B, use Eqn. 2 for temperature, pressure, and relative humidity observations as numbered.

$$d_n = \frac{X_1 - S_1 + X_2 - S_2}{2} \quad \text{Eqn. (2)}$$

3.2 Calculate the mean value for the observed measured differences, \bar{d} .

3.3 Calculate the correction for each instrument parameter (temperature, barometric pressure, relative humidity as applicable), C_x , using the correction for the reference standard, C_s , taking care to ensure the correct sign and reference value is used. Note: some corrections, especially in thermometry, are not additive to the nominal value.

$$C_x = C_s + \bar{d} \quad \text{Eqn. (3)}$$

4 Measurement Assurance

4.1 Three replicate comparisons are performed during the measurement process to calculate a replicate standard deviation.

4.2 Replicate standard deviations are tracked on a standard deviation chart for this process and the replicate process standard deviation are pooled over time. Calibration repeatability is evaluated against the limits on the chart and assessed based on the limits of uncertainty needed for subsequent calibration and use in the laboratory. Where repeatability and final uncertainties are too large, a better calibration method or calibration source are essential.

4.3 Where a check standard instrument is available, values for that instrument may be tracked over time. However, if instruments are not identical in type and resolution, the variability of a check standard may not be a suitable duplication for an unknown standard to use the standard deviation of the check standard for the process repeatability. The value of the check standard may provide a suitable check on the stability of measurement results over time.

5 Assignment of Uncertainty

5.1 Uncertainty for the standard, u_s .

The uncertainty for the standard is obtained from the calibration certificate for the reference standard that is used for this calibration. Determine the degrees of freedom for the standard by reviewing the calibration certificate. If a coverage factor of 2 is stated, an estimate of 500 degrees of freedom may be used for the standard (this is an estimated value and other reasonable estimates may be used).

5.2 Uncertainty for the process, s_p .

The uncertainty for the standard deviation of the process is obtained from the replicate standard deviation.

Since the degrees of freedom are very small for this calibration (2), a higher coverage factor may be needed (See Appendix A in NISTIR 6969, SOP 29). A greater number of replicate observations may be obtained for an equal number of days, e.g., 7 observations obtained over 7 days, or 10 observations obtained over 10 days to increase the degrees of freedom for this component.

Use a pooled replicate standard deviation where possible. A pooled value of the replicate standard deviation will provide greater degrees of freedom to be used for calculating the coverage factor for the uncertainty.

Where the standard deviation is less than the resolution of the instrument, use an uncertainty for the process, u_p , of the larger of either the calculated standard deviation, s_p , or the value calculated from the following formula. The laboratory may also round up to the nearest resolution of the instrument.

$$u_p = \frac{\text{resolution}}{\sqrt{3}} \quad \text{Eqn. (4)}$$

5.3 Uncertainty for other components, u_o .

Where instrument history or stability information are available, additional components should be considered in the evaluation of the uncertainty. The estimate of drift during a calibration interval may be treated as a rectangular distribution. While this will increase the uncertainty, it will provide a more realistic assessment of uncertainty to be used when evaluating the suitability of this procedure.

Table 2. Example Uncertainty Budget Table.

Uncertainty Component Description	Symbol	Source	Typical Distribution
Uncertainty of the standard used as a reference (temperature, pressure, relative humidity)	u_s	Calibration certificate	Expanded divided by the coverage factor, k
Standard deviation from replicate measurements	s_p	Standard deviation from replicate measurements, Pooled standard deviation where data is available from similar instruments	Normal
Standard deviation from replicate measurements – when standard deviation is less than resolution	u_p	Estimated process uncertainty based on resolution of the instrument being calibrated	Rectangular or rounded up to nearest resolution
Estimates of drift	u_o	Historical evaluation of stability in the instruments (where available)	Rectangular

5.4 Calculate the combined and expanded uncertainty following the procedure in NISTIR 6969, SOP 29. Use appropriate effective degrees of freedom using the Welch-Satterthwaite equation as noted in NISTIR 6969, Section 8 or from the Guide to the Expression of Uncertainty in Measurement.

5.5 Evaluate the expanded uncertainty.

Determine whether the measurement results and uncertainties will meet requirements for corrections needed for calibrations (e.g., per SOP 2), for monitoring laboratory conditions, or for other intended application and use.

6 Certificate

Report results according to SOP 1, ISO/IEC 17025, and applicable customer requirements. Report the mean values of the temperature, pressure, relative humidity (as appropriate). Include the range of environmental conditions observed during the calibrations and report the estimated calculated uncertainties. Report the reference calibration certificate and date for the instrument used as the standard. Include a suitability statement to the effect of whether the calibration may be used for calibration corrections and/or for monitoring laboratory environmental conditions.

Note: If this is an internal calibration and/or not covered by the scope of accreditation or recognition, follow accreditation body requirements regarding any additional statements that may be required when such measurements are not covered by the Scope.

Appendix A – Data Sheet

Laboratory data and conditions:

Operator		Laboratory Conditions	Before	After
Date Range		Temperature		
Replicate standard deviation of the process, from standard deviation chart, s_p		Pressure		
Degrees of Freedom		Relative Humidity		

Standard(s) data:

ID	Nominal ^a	Correction ^b	Measurement Units	Expanded Unc: From cal. certificate	Unc. <i>k</i> factor	Unc. Units
<i>S</i> – temperature						
<i>S</i> – pressure						
<i>S</i> – relative humidity						
^a selected reference value from the calibration certificate nearest to normal laboratory conditions.						
^b taking care to determine directionality as appropriate						
Identification of the standards, serial number:						
Identification of the standards, calibration source:						
Identification of the standards, date of calibration:						

Measurement Observations, Option A (Record Units in Column Headings):

Dates	S_t _____	X_t _____	S_P _____	X_P _____	S_{RH} _____	X_{RH} _____
1						
2						
3						

Measurement Observations, Option B (Record Units in Column Headings):

	Temperature_____				Pressure_____				Relative Humidity_____			
Dates	S_1	X_1	X_2	S_2	S_1	X_1	X_2	S_2	S_1	X_1	X_2	S_2
1												
2												
3												