Specific Accreditation Criteria

ISO/IEC 17025 Application Document

Calibration - Annex

Temperature metrology

May 2022

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Temperature Metrology

Purpose

In addition to the *ISO/IEC 17025 Standard Application Document* (SAD) and the accompanying Calibration - Appendix, this document provides interpretative criteria and recommendations for temperature metrology for both applicant and accredited facilities.

Facilities must comply with all relevant documents in the NATA Accreditation Criteria (NAC) package for Calibration (refer to *NATA Procedures for Accreditation).*

For ease of use and to avoid fragmentation of the information, the relevant clause numbers of ISO/IEC 17025 have not been included.

Criteria and recommendations for all types of measurement equipment

Where a facility uses a liquid calibration bath for testing temperature sensors at temperature levels sufficiently high for the liquid (e.g. silicone oil) to fume, a fume exhaust system should be installed above the bath.

In the situation where a fluidised bed bath or a salt bath is used, adequate measures should be made to contain the heated medium to prevent hot particles escaping towards the test operator.

Electric furnaces for calibration purpose should be installed in a manner to obviate AC pick up which may influence the test data.

Methods with examples of uncertainty of measurement focussed on Heat and Temperature Measurement can be found in *NMI Monograph 1, Uncertainty in Measurement: The ISO Guide*, RE Bentley.

The term “reference thermometers” refers to a thermometer which is reserved for the calibration or checking of working thermometers. They should not be routinely used for client calibrations or measurements. In general, the uncertainty of calibration of a reference thermometer must be no more than $1/5^{th}$ of the uncertainty of calibration required of the working thermometer (refer to the requirement for *Review of requests, tenders and contracts* as stated in the Calibration Appendix).

Ice-points must be made with de-ionised or distilled water. Uncertainties of 20 mK or less must be supported by regular measurement of the conductivity of the water. Uncertainties of 4 mK or less require the ice-point to be regularly checked against a calibrated water-triple-point cell.

Reports must explicitly state the temperature scale on which the temperature is reported, this will normally be ITS-90. However, it may also be appropriate to specify IPTS-68 (e.g. for historical compatibility reasons) or thermodynamic temperature (e.g. lamp colour or distribution temperatures).

Criteria and recommendations for specific types of measurement equipment

Apparatus for fire tests

The critical dimensions of the apparatus must be measured and recorded to establish compliance with the requirements of AS 1530.1, .3 and .4 on *Methods for fire tests on building materials, components and structures.*

Automatic reference junctions

Where automatic reference ice-point junctions are used in place of an ice pot, their accuracy and stability of performance must be assessed. For thermocouple logging systems used for field work outside of a facility (at ambient temperatures outside of 20°C to 25°C) the effectiveness of their ACJC is to be tested at ambient temperatures appropriate to the field work being undertaken.

Calibration baths and furnaces

Baths and furnaces used for calibration purposes must have their temperature uniformity characteristics determined over the temperature range for which accreditation is required. In addition, the effects of sample loading and thermal losses on performance should be assessed as is applicable to the application. A record covering the testing techniques used and commenting on the suitability of baths and furnaces for calibration purposes is to be retained.

Dry block calibrators

When calibrating dry-block calibrators, axial uniformity must be assessed over a distance of 20 mm and reported or included as an uncertainty component. The reference sensors should be selected for minimal heat conduction error (refer to conduction errors in this document). The uniformity test is to be performed at least at the maximum and minimum temperatures. The report of a dry block calibrator should identify the relevant insert and the dimension of the probe.

When dry-block calibrators are used as a temperature source for sensor calibrations, a “pull up 20 mm” test of the device under test (DUT) and reference needs to be performed at (at least) one temperature, and included as a component in the uncertainty calculations (e.g. propagated proportionally to temperature).

Additional guidance on the calibration of dry block calibrators may be found in *EURAMET cg-13, Calibration of Temperature Block Calibrators*.

Dry block heaters

As dry block heaters may be used for a variety of applications, calibration of these devices requires consideration of the intended use, including but not limited to:

- at a minimum, measurements at the highest and lowest temperatures of intended use;
- measurements made with temperature sensors touching the bottom of the thermometer well or sample enclosure;
- sensors should be selected for minimal heat conduction error (refer to conduction errors in this document);
- stability and uniformity are to be measured over a period of at least one hour with measurements taken at least once every 10 minutes;
- if used with sample enclosures, it is recommended that uniformity measurements be made using thermometers immersed in close-fitting sample enclosures containing oil. The oil level within the sample enclosure should not exceed the height of the well in the modular block. A sufficient number of sample sites must be spatially distributed over each block to measure gradient maximums.

Reports are to identify the following items as applicable:

- the mean block and indicated temperatures measured at steady-state condition with reporting of the temperature correction to the indicated temperature;
- magnitude of temperature instability;
- magnitude of temperature non-uniformity;
- calibration conditions including details of the blocks and their relative position, the number and site/s of the temperature measurements, user-set calibration offsets (if any), loading of wells, and if used, details of the sample enclosures and fluid.

Conduction errors

When calibrating probe thermometers, the error due to heat conduction along the probes must be taken into account in the uncertainty. An immersion sensitivity test may be performed to check for this effect. Conduction errors for probes immersed more than 200 mm into a fluid bath or 250 mm into a tube-furnace can generally be assumed to be negligible, and the corresponding enclosure uniformity value alone used as the transfer error. Probes calibrated in dry-well calibrator or immersed less than this must have an immersion test performed on each DUT. This consists of observing the temperature change when the probe is withdrawn a further 20 mm. This test should be performed at the extreme temperature (T_{max} or T_{min} of the bath or furnace) or at a minimum, the extremes of application and propagated proportionally to ($T-T_{amb}$).

Environmental enclosures (e.g. environmental chambers, conditioning rooms, calorimeter rooms)

The spatial and temporal uniformity of the enclosures for all required parameters must be determined. Single point measurements are not acceptable.

Enclosures are to be tested to ensure that they comply with the requirements of the test procedures for which they are used.

Valid methods such as those published in *IEC 60068.3.5, IEC 60068.3.6* and *EURAMET cg-20, Calibration of Temperature and/or Humidity Enclosures* may be used for the testing of all enclosures and chambers. Medical refrigeration equipment used for the storage of blood and blood products must be calibrated following *AS 3864.2*.

Records of characterisation of the environmental enclosure must be maintained.

The loading state of the enclosure must be specified in the report.

Ovens and furnaces

Ovens and furnaces are to be calibrated against a suitable method valid for the application such as those listed in *IEC 60068 Part 3* and the *EURAMET guide cg-20* and the following must be taken into account:

- to minimise error due to annealing, heat exposed wire (for temperatures greater than 400°C) should not be used in the temperature gradient zone in subsequent tests (e.g. cut new wire or move new wire into the gradient zone);
- the furnace or oven must be allowed to stabilise for at least 1 hour, or 5 control cycles, prior to performing the calibration;
- the number of thermocouples must be greater than or equal to that required by the method;
- the use of MIMS type thermocouples is strongly recommended. However, if ceramic or fibre-glass sheathed wire is used, it is essential to ensure the wire cannot be contaminated by vapours from the binder. In this case, where possible, the thermocouple passes through a vent rather than the oven door and if there is evidence of contamination to the wire in the gradient zone the test should be repeated.

Autoclaves

The homogeneity of the thermocouples feeding through to the chamber should be checked at regular intervals as this is usually also the EMF generating temperature gradient zone. This can be achieved by placing thermocouple tips in an ice-point, heating the feed-through with a hot air gun and confirming that changes are negligible.

Thermocyclers

PCR thermocyclers use a variety of technologies to achieve rapid heating and cooling sequences across a number of tubes. The accuracy of temperature and hold time, the magnitude of overshoots and undershoots, the uniformity of temperature, and heating and cooling rates across the thermal block are critical aspects to the performance verification of thermocyclers.

Due to the dynamic performance of thermocyclers, they must be verified in a dynamic way. It must be ensured that the reference equipment used for the calibration of thermocyclers has suitable evidence of metrological traceability and is fit for purpose. The following must be taken into account::

- each sensor is to have a temperature uncertainty no greater than 1/5th of the uncertainty of calibration required for the thermocycler under test;
- the dynamic response time of the temperature sensors must be suitably rapid to measure the rise and fall time as well as set point over/undershoot. This can be assessed by exposing the sensor to a typical step change in temperature e.g. by quickly withdrawing the sensor to ambient air from a temperature of 50 °C or above, or vice versa. The response time to measure 63% of the step change should be below 2 seconds;
- simultaneous (or near simultaneous) and continuous logging of multiple well temperatures at each measurement site during the test interval (where the test interval is one complete cycle of the thermocycler). The sensor sampling/logging frequency must be 1 Hz or more. The time accuracy of the logging system must be better than 1 in 10^5;
- the resolution is appropriate for use, e.g. 0.01°C.

A thermocycler calibration system will thermally load the thermocycler in terms of thermal mass and thermal conductivity (heat leaks) differently to a set of PCR samples. Consequently different calibration equipment may lead to different results for the same thermocycler. The facility must consider:

- the magnitude of systematic error due to the combination of the calibration system and the thermocycler;
- assessment of the systematic effects of loading by the calibration system must be undertaken. This may be performed by comparing the calibration results from the calibration system to that of a basic method with known small uncertainties. For example, a 0.1 mm diameter calibrated thermocouple wire placed into a sample tube containing a drop of oil. An error of less than 1/5th of the uncertainty of calibration required for the thermocycler under test is considered acceptable. This term would be included in the reported uncertainty of measurement.

Thermocyclers often have a heated lid preventing condensation and evaporation. Opening the heated lid can influence the calibration results therefore, when possible, calibrations should be performed with the lid closed to replicate in-use conditions.

Temperature uniformity must be reported across the thermal block and as such the number of sensors be selected proportionate to the number of wells with consideration to the size of the block. The number of sensors should be at least 12.5% of the number of wells for thermocyclers with up to 96 wells, subject to a minimum of 5 sensors for thermocyclers with 8 or more wells, and a minimum of 12 sensors for thermocyclers with more than 96 wells. Sensors must be placed in the four corner positions and spatially and evenly distributed over the middle and edges of the block.

The report must state if the calibration was performed with the heated lid opened or closed and if closed during calibration, report the heated lid temperature. If during calibration the lid is open, the report must indicate that temperatures reported may be different to that during normal use with the lid closed.

In order to suitably verify the performance of a thermocycler the following items should be reported pending the application and requirements of the thermocycler:

- the mean block temperature measured after a 15s to 30s stabilisation period and the temperature correction to the indicated temperatures at the lowest and highest temperature settings, a low- to mid-range setting and a mid- to high range setting to match the in-use temperature range. For example, 15°C, 55°C, 72°C and 95°C may suit most users or 4°C, 55°C, 72°C and 98°C if the customer intends to use the device at the cycler's full range;
- the stabilisation period;
- the temperature heating and cooling rates and set point overshoot/undershoot for each sensor which may be stated in a graphical form during a typical cycle;
- the maximum overshoot and undershoot at each measurement point;
- time at temperature after overshoot/undershoot for each temperature set;
- temperature uniformity across the thermal block.

All thermometers

The report must state whether the reported uncertainty includes a component for linearity/interpolation error, or is valid only at the reported calibration points.

The report must state any annealing procedure applied to the thermometers prior to calibration.

Liquid-in-glass thermometers

Temporary depression must be measured by measuring the correction at a reference temperature (usually the ice-point) before and after exposure to temperatures away from ambient and any shift included as a component in the uncertainty calculations.

If the thermometer is for >0°C usage and the ice-point mark is not provided on the scale, the reference temperature should be the lowest cardinal temperature.

Platinum resistance thermometers

Hysteresis is usually the largest uncertainty component for resistance thermometers, and needs to be assessed for each DUT.

A determination of the hysteresis must be evaluated. This may be undertaken by repeat measurements at the ice-point before and after each temperature excursion (e.g. before and after cooling to -40°C or heating to 100°C) and any difference included in the uncertainty as a hysteresis (short term drift) component.

For low uncertainty sensors used far from ambient (e.g. less than 0.2°C uncertainty and <-40°C and/or >100°C), the ice-point hysteresis test is insufficient by itself. After calibration at the most extreme temperature points (Tmin) and (Tmax), a determination of the hysteresis may be undertaken by repeat measurements at or near Tmin/2 and Tmax/2 on the way back to zero.

For example, a PRT calibrated from -90°C to 0°C could use the following sequence: 0°C, -45°C, -90°C, -45°C, 0°C, and a PRT calibrated from 0°C to 650°C may use the following sequence: 0°C, 100°C, 200°C, 300°C, 400°C, 500°C, 600°C, 650°C, 300°C, 0°C.

Vapour pressure thermometers

The effect of stiction must be determined by taking readings at both rising and falling temperatures for at least one point in the range and included as a component in the uncertainty calculations.

The report must state the orientation of the probe (e.g. vertical or horizontal).

Thermocouples

It is important to note that the EMF in a thermocouple is produced in a temperature gradient and not at the thermocouple tip and thus the effects of inhomogeneity must be considered along with the following:

- thermocouple reports must state the EMF-to-T reference function used (e.g. ASTM…);
- the inhomogeneity of a thermocouple must be included as a component in the uncertainty calculations, For new thermocouples a default value of 0.1% for base metal and 0.02% for rare-metal thermocouples should be used, For used thermocouples it must be measured, for example, by measuring the correction over a range of at least 100 mm in immersion depths;
- the inhomogeneity uncertainty component can be propagated proportionally to temperature;
- because base metal thermocouples can experience significant drift during calibration, the calibration procedure for base metal thermocouples must require measurements over at least a ½ hour period at mid and highest temperature of calibration and the variation obtained included as an additional uncertainty component (drift);
- ceramic or fibreglass insulated base metal wire test method must specify that air can circulate freely to ensure binder vapours do not contaminate the thermoelements;
- for MIMS thermocouples with a head, where a junction involves different wires, the calibration procedure must estimate any error due to different wires

in the MIMS and extension leads (e.g. by putting the sensor in an ice-point and noting any change when the head is warmed or cooled);
- for the calibration of reels of thermocouple wire, inhomogeneity in the reel must be evaluated. The estimated inhomogeneity may need to be revised if the measured variation between samples is significantly different to that estimated (could be lower or higher). In this case additional homogeneity measurements may be required.

Digital temperature indicators

Reports must state the values of any internal coefficients accessible to the user (e.g. a, b coefficients of ITS-90 PRT equations).

The following are to be considered as applicable to the sensor type:

- an uncertainty component for the temperature coefficient of the internal ACJC must be included in the uncertainty calculations. If it is not explicitly measured, a default value of 0.1°C per °C variation in ambient is typical;
- the report must state if the calibration was performed with internal or external ACJC. If an external ACJC is used, a calibrated thermocouple together with a reference temperature such as an ice-point must be used;
- if the calibration is performed by electrical simulation, the report must state explicitly that it is an electrical simulation calibration and that any sensor must be separately calibrated.

Radiation thermometers (e.g. pyrometers)

The Size-of-Source-Effect (SOSE) is usually the largest uncertainty component in calibration of these types of instruments, and must be explicitly measured for each DUT during calibration at least at one temperature. This can be achieved by recording the change in indicated reading if:

- the diameter of an aperture in front of the source is reduced by 10mm; or
- the calibration distance is changed by 25%.

The report must state the diameter of the radiation source used and its distance from the DUT.

Because of differences in the spectral responsivity of DUT and reference radiation thermometer, comparison calibrations against grey-body sources require an additional component in the uncertainty calculations.

When defining customer requirements, consideration is to be given to the emissivity or emissivities applicable to the radiation thermometer’s end use. Facilities must demonstrate that they are capable of calculating and applying corrections if calibration is required at emissivities other than the calibrated emissivity of the reference thermometer. The CMC stated in the facility’s NATA Scope of Accreditation will include any fixed emissivities or the range of emissivities covered.

Surface probes and calibrators

The surface probe sensor needs to be physically reheated several times for at least one temperature (typically the highest) to assess contact variability. The measured variation should be propagated proportionally to temperature as an additional uncertainty component.

For calibration of surface probes, the report must identify the surface used for the calibration and the nature of the ambient air flow (e.g. “calibrated on a polished aluminium plate in still air”).

Humidity

The CMC in the Scope of Accreditation will include the temperature (range) to which the humidity is reported with corresponding uncertainty of measurement across humidity range and temperature range.

Users of humidity measuring equipment must ensure the temperature component is calibrated to suit the intended use.

The unit to be used for values of relative humidity expressed as a percentage is “%rh”. The term Relative Humidity may be abbreviated to RH.

Hysteresis is the maximum difference in sensor response when exposed to the same reference humidity/temperature condition following recent exposure to a higher or lower humidity at the same temperature. It is recommended to calibrate impedance-type humidity sensors over the range of use and not wider, as the largest hysteresis loops are experienced when the sensor is exposed to the widest range of relative humidities, especially to levels of humidity above 70 %rh.

When calibrating an impedance-type humidity sensor over a wide range, evaluation of the hysteresis may be made by repeating measurements at mid-range humidity between 35 %rh and 65 %rh, before and after each low and high humidity excursion. Appropriate soak time between measurement points must be considered, taking into account the manufacturer’s recommendations on the use of the device. This evaluation of the hysteresis can be considered as a short-term drift contribution to the uncertainty calculation. The report must include a statement that the uncertainty includes a component for the effect of hysteresis.

When evaluating the hysteresis characteristic, consideration should be given to the calibration temperature demonstrating the largest effect; typically this would be the highest calibration temperature. If the evaluation of hysteresis can not be performed, a value of 0.3 %rh to 1 %rh is considered appropriate for a sensor soaked at 70 %rh to 95 %rh, and 0.2 %rh if the highest humidity is below 70 %rh.

A minimum of three humidity calibration points spanning the customer’s humidity range of use is recommended to be conducted during a hygrometer calibration.

The recommended reference equation for the calculation of vapour pressures above water and ice, used in the calculation of relative humidity, is the saturation vapour pressure formulation given by Sonntag. The formula and coefficients can be found in the Appendix of *NMI Monograph 8: Humidity and its Measurement*.

Aqueous salt solutions may be used for the calibration of humidity sensors, using a calibrated reference hygrometer for traceability. The device containing the solution used for maintaining a stable humidity environment (hygrostat), must be operated in a tightly temperature-controlled environment with no draughts and no direct sunlight, and in accordance with the manufacturer’s recommendations on the use of the device. Sufficient time must be allowed for the sensors, air and solution within the measuring space of the hygrostat to reach temperature equilibrium. As aqueous salt solutions are very sensitive to ambient humidity leaks, an air-tight seal is required.

The report must state the temperature range for which the humidity range is valid.

Electrical calibration of temperature indicators, such as digital multimeters and digital temperature indicators

The calibration process for devices must include:

- if internal ACJC is used, a calibrated thermocouple, together with a temperature reference, such as ice-point;
- the V=>T or R=>T conversion software within the instrument must be validated at several points over the calibration range. For thermocouple sensors this includes separately checking each of the thermocouple ranges;
- an uncertainty contribution to allow for non-linearity of the resistance or voltage measurement scale of the DUT (e.g. manufacturers specification), when the DUT is calibrated as an ohmmeter or a voltmeter with a subsequent validation of the R=>T, V=>T conversion. To achieve a lower uncertainty, several points over the range must be checked.

The report must include the following:

- for thermocouple ranges, whether the calibration is performed with external or internal ACJC;
- which V=>T, or R=>T conversion equations have been selected (e.g. ASTM table XXX ref. XXX. IEC-60751, ITS-90 SPRT reference equations, etc.);
- any internal coefficients stored as part of the conversion equations (e.g. a,b,c coefficients in ITS-90 SPRT reference equations);
- the temperature scale the calibration of the device is based on (e.g. IPTS-68, ITS-90, etc.);
- explicitly state that the calibration is an electrical simulation only.

References

This section lists publications referenced in this document. The year of publication is not included as it is expected that only current versions of the references shall be used.

Standards

AS 1530.1	Methods for fire tests on building materials, components and structures - Combustibility test for materials
AS 1530.3	Methods for fire tests on building materials, components and structures - Simultaneous determination of ignitability, flame propagation, heat release and smoke release
AS 1530.4	Methods for fire tests on building materials, components and structures - Fire-resistance test of elements of construction
AS 3864.2	Medical refrigeration equipment - For the storage of blood and blood products - User-related requirements for care, maintenance, performance verification and calibration
IEC 60068.3.5	Environmental testing - Supporting documentation and guidance - Confirmation of the performance of temperature chambers
IEC 60068.3.6	Environmental testing - Supporting documentation and guidance - Confirmation of the performance of temperature/humidity chambers
ISO/IEC 17025	General requirements for the competence of testing and calibration laboratories

NATA Publications

NATA Accreditation Criteria (NAC) package for Calibration

General Accreditation Criteria	ISO/IEC 17025 Standard Application Document
Specific Accreditation Criteria	ISO/IEC 17025 Standard Application Document, Calibration - Appendix

Other Publications

EURAMET cg-13	Calibration Guide, Calibration of Temperature Block Calibrators
EURAMET cg-20	Calibration Guide, Calibration of Temperature and/or Humidity Controlled Enclosures
NMI Monograph 1	Uncertainty in Measurement: The ISO Guide, RE Bentley
NMI Monograph 8	Humidity and its Measurement

Amendment Table

The table below provides a summary of changes made to the document with this issue.

Section or Clause	Amendment
Whole document	General editorial revision.
Thermometers	The guidance for annealing or conditioning moved from LIG types to all types of thermometers
Humidity	Clarification of units to %rh
Humidity	Added guidance on calibration method including evaluation of hysteresis
Humidity	Added guidance on the use of salt solutions
References	Added reference to NMI Monograph 8