Specific Accreditation Criteria
Calibration ISO/IEC 17025 Annex

Temperature metrology

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Temperature Metrology

This document provides interpretative criteria and recommendations for the application of ISO/IEC 17025 in the field of Calibration for both applicant and accredited facilities.

Applicant and accredited facilities must also comply with ISO/IEC 17025 and the NATA ISO/IEC 17025 Standard Application Document (SAD) and the NATA Calibration ISO/IEC 17025 Application Document. The clause numbers in this document follow those of ISO/IEC 17025. However since not all clauses require interpretation, the numbering may not be consecutive.

This appendix is divided into two sections; the first provides additional guidance and recognition of the general requirements that are applicable to all types of measurements in this discipline, while the latter provides additional guidance and recognition of the requirements that are specific to individual types of measuring equipment. For ease of use and to avoid fragmentation of the guidance, the clauses of ISO/IEC 17025 have not been applied.

General requirements

Fume exhaust system

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 must 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.

Calibration methods and method validation

Note: Methods with examples of uncertainty of measurement focussed on Heat and Temperature Measurement applications include:

- *Uncertainty in Measurement:* The ISO Guide – RE Bentley, NMI Monograph 1
- *Applying the ISO Guide to the Calculation of Uncertainty: Temperature* – RE Bentley, NML Publ. No. TIP P1358

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, should be no more than 1/5th of the uncertainty of calibration required of the working thermometer.

Calibration 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 is IPTS-68 (e.g. historical compatibility reasons) or thermodynamic temperature (e.g. lamp colour or distribution temperatures).

Ice-points must be made with de-ionized 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.

Specific requirements for 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 should be assessed. Thermocouple logging systems used for field work outside of a facility (at temperatures outside of 20°C to 25°C) should have the effectiveness of their ACJC 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 bath performance should be assessed. A detailed record covering the testing techniques used and commenting on the suitability of baths for calibration purposes must be available.

Dry block calibrators (calibration and use of)

In calibration of dry-block calibrators, the uniformity must be assessed over a distance of 20 mm and reported or included as an uncertainty component.

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).

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

Conduction errors:

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 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.

The enclosures must be tested to ensure that they comply with the requirements of the test procedures.

The method in AS 2853 Performance of Heated Enclosures may be used for the testing of all enclosures and chambers (ovens, baths, furnaces, incubators, freezers, etc.).

The results of characterisation of the environmental enclosure must be available for examination during an assessment.

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

Ovens and Furnaces

Ovens and furnaces would normally be calibrated against a standard such as AS 2853 or similar:

- 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 AS 2853. Any departure from this must be explicitly stated on the report and the report cannot state that the testing has been performed according to AS 2853;
- 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, the test method should specify that if possible the thermocouple passes through a vent rather than the oven door, and that if there is evidence of contamination to the wire in the gradient zone the test should be repeated.

Autoclaves

The homogeneity of the thermocouples is the feed-through to the chamber should be regularly checked, 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. A traceable calibration is seen as desirable for quality-control and method portability and to numerically verify the performance of the device.

Due to the dynamic performance of thermocyclers, they must be verified in a dynamic way. Facilities must ensure that the reference equipment used for the calibration of thermocyclers has suitable evidence of metrological traceability and is fit for purpose, giving consideration to:

- 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, the time response 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 should be 1 Hz or more. The time accuracy of the logging system should be better than 1 in 105;
- the resolution should be 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, should be taken into account for each combination as part of the calibration method validation process;
- assessment of the systematic effects of loading by the calibration system 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 this lid closed to replicate in-use conditions.

The test method will report temperature uniformity across the thermal block and as such the number of sensors should 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, report the heated lid temperature. If the lid is open, the report must clearly state this and that temperatures reported may be different to that during normal use with the lid closed.

Additionally in order to suitably verify the performance of a thermocycler, reports should at least include the following items:

- 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;
- temperature uniformity measured after a 15s to 30s stabilisation period;
- 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 error, or is valid only at the reported calibration points.

Liquid-in-glass thermometers

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

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

Platinum resistance thermometers

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

A measurement of the calibration at the ice-point should be made before and after each temperature excursion (e.g. before and after oil bath measurements etc.)

For low uncertainty sensors used far from ambient (e.g. less than 0.2°C uncertainty AND <-40°C or >100°C), the ice-point hysteresis test is insufficient, and a measurement at $T_{max}/2$ on the way back to zero must be performed.

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 should 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, and the variation obtained included as an additional uncertainty component (drift);
- the calibration procedure for ceramic or fibreglass insulated base metal wire must specify that air can circulate freely to ensure binder vapours do not contaminate the thermoelements;
- for MIMs thermocouples with a head junction, 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

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

In addition to the requirements for thermocouples (above) the following also must be considered:

- 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;

- aperture in the 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.

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 state 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

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

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

A calibration 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 Automatic Cold Junction Compensation 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 and other references

AS 1000 *The International System of Units (SI) and its application*

AS 1530.1 *Methods for fire tests on building materials, components and structures - Combustibility test for materials*

AS 2853 *Enclosures – Temperature Controlled-Performance testing and grading*

EURAMET cg-13 *Calibration of Temperature Block Calibrators*

Uncertainty in Measurement: The ISO Guide – RE Bentley, NMI Monograph 1

Applying the ISO Guide to the Calculation of Uncertainty: Temperature – RE Bentley, NML Publ. No. TIP P1358

Amendment Table

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

Section or Clause	Amendment
New document	This document represents a direct adoption of the former Calibration Application Document Appendix E. The document has been reviewed and updated to reflect the new accreditation criteria documentation structure.