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Calibration of Temperature Block Calibrators

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Calibration Guide

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CALIBRATION OF TEMPERATURE BLOCK CALIBRATORS

Purpose

This document has been produced to enhance the equivalence and mutual recognition of calibration results obtained by laboratories performing calibrations of temperature block calibrators.

Authorship and Imprint

This document was developed by the EURAMET e.V., Technical Committee for Thermometry.

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Guidance Publications

This document gives guidance on measurement practices in the specified fields of measurements. By applying the recommendations presented in this document laboratories can produce calibration results that can be recognized and accepted throughout Europe. The approaches taken are not mandatory and are for the guidance of calibration laboratories. The document has been produced as a means of promoting a consistent approach to good measurement practice leading to and supporting laboratory accreditation.

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1 SCOPE

1.1 This Guideline applies to temperature block calibrators in which a controllable temperature is realized in a solid-state block with the aim of calibrating thermometers whose sensing element is inserted into the borings. A temperature block calibrator comprises at least the block located within a temperature-regulating device, and a temperature sensor with indicator (the built-in controlling thermometer) to determine the block temperature.

Warning:

The calibration must not be confused with the characterisation of the device. The characterisation consists in determining the thermal behaviour of the device (spatial and temporal uniformity). The calibration consists in establishing the relation between the temperature generated at a given place (usually a volume) of the device (unambiguously specified) and the value read on the temperature indicator. A previous characterisation of the device is necessary for associating the uncertainties of the calibration.

1.2 This Guideline is valid in the temperature range from -100 °C to +1300 °C. The temperature ranges stated by the manufacturer shall not be exceeded.

2 CALIBRATION CAPABILITY

2.1 This Guideline is only applicable to temperature block calibrators that meet the following requirements:

The borings used for calibrations shall have a zone of known temperature homogeneity (in the following referred to as measurement zone), whose position is exactly specified, and suitable for the thermometer to be calibrated. The measurement zone will in general be at the lower end of the boring. If the measurement zone is situated at another place, this shall explicitly be stated..

2.2 It shall be ensured that calibration is possible under the following conditions:

In the temperature range from –100 °C to +660 °C, the inside diameter of the boring or, if present, a bushing inserted to adapt the diameter of the boring, may be at most 0.5 mm larger than the outside diameter of the thermometer to be calibrated; in the temperature range from +660 °C to +1300 °C, this value may be at most 1.0 mm. As an alternative, an equally good or better thermal contact may be established by suitable heat-conveying means or medium, such as an oil, subject to compatibility with the materials of the block and the thermometer, and the temperature of use. The thermal contact is a vital uncertainty contribution at very high precision calibrations and must be evaluated, especially if no heat-conveying means are used.

In all cases the calibration setup (thermometers and dry block calibrator) must be designed that conduction of heat along their length does not give rise to excessive error and uncertainty (especially at high temperatures) so. This is usually one of the dominant source of uncertainty in the uncertainty budget of a thermometer calibration.

3 CHARACTERISATION

3.1 General

3.1.1 When a temperature block calibrator is used or calibrated, the characteristics of the temperature distribution in the measurement zone (defined in sections 3.2 to 3.5) must be investigated and documented.

3.1.2 All investigations shall be carried out under the measurement conditions stated in sections 2.1 and 2.2.

3.1.3 If adapter bushings are required to comply with the requirement of section 2.2, these preferably will be made of the material proposed by the manufacturer.

3.1.4 If the temperature block calibrator has one or several borings in which a bushing is used, it is to be agreed with the customer which bushing (or bushings) is (are) to be used. If bushings are used , the diameters are to be investigated in the same way as the borings in the temperature block calibrator. Unambiguous marking of the bushings is required.

3.1.5 The thermometer used for the investigations according to sections 3.2 to 3.4 (test thermometer) need not be calibrated, as these tests are performed to measure the temperature differences. The sensitivity at the measuring temperature shall, however, be known with a sufficiently small uncertainty. The sensitivity can usually be taken from the respective standard and is to be checked by a control measurement (possibly at a different temperature). The stability of the thermometers used during the characterisation shall be tested.

3.1.7 The investigations described in the following Sections 3.2 to 3.5 are to be carried out.

3.2 Axial temperature homogeneity along the borings in the measurement zone

The influence of the temperature distribution in the measurement zone along the borings (axial temperature distribution in each boring) is to be determined in such a way that it can be taken into account in the uncertainty budget of the calibration. Potential methods are presented in Annex B.

The necessary investigations are to be carried out at the operating temperature showing the greatest difference from the ambient temperature (both positive and negative). If it is assumed that the influence of the temperature distribution at other operating temperatures can be estimated by linear interpolation, this must be checked by tests at additional temperatures.

3.3. Temperature differences between the borings

The greatest temperature difference occurring between the borings is to be determined. At least the measurement of the temperature difference between (opposite) borings situated at as great a distance from each other as possible is to be determined. To eliminate the influence of temperature variations with time, the temperature differences with respect to an additional test thermometer in the temperature block calibrator could be determined.

3.4 Influence upon the temperature in the measurement zone due to different loading

In the case of use of several borings in the dry block, more detailed investigations into the influence on the temperature in the measurement zone due to different loadings can be made upon customer request. In this case, the results for loading with only one thermometer and with all borings loaded are compared. Loadings with thermometers can be simulated by loadings with metal or ceramic tubes. The measurements are to be carried out at least at the temperature with the largest temperature difference from the ambient temperature (both positive and negative).

3.5 Stability with time

Depending of the temperature, a sufficient time to reach the thermal equilibrium must be reserved in order to make proper measurements. This point is particularly important in case of on-site use. The maximum range of temperatures indicated by a sensor in the measurement zone over a at last a 30 minute period, when the system has reached equilibrium, shall be determined.

Measurements are to be performed at the highest and at the lowest test temperature

3.6 Temperature deviation due to heat conduction

Note that the thermometer used for the characterization may influence thermally the area under calibration due to heat losses, depending on the sensor design.

4 CALIBRATION

The deviation of the indication of the built-in (or optional external) controlling thermometer from the temperature in the measurement zone must be established by a calibration. If the control of the block calibrator is set from either the external or internal thermometer – this must be remarked and agreed with the customer.

The temperature in the measurement zone of the temperature block calibrator is determined with a standard thermometer, which is traceable to national standards.

4.1 Measurements

The calibration is performed using the standard thermometer in the central boring or in a particularly marked boring. The calibration points must be define with the customer.

At each calibration point, two measurement series are carried out, in which the average for the deviation of the indication of the built-in controlling thermometer from the temperature in the measurement zone is determined. The sequence of the calibration point is done for one measurement series at increasing temperatures and for the other at decreasing temperatures. However, at least two measurement series are to be recorded, between which the operating temperature of the calibrator is changed.

The values measured in the series at increasing and decreasing temperatures are averaged for each calibration point. The calibration result (deviation of the temperature measured with the standard thermometer from the indication of the calibrator) is documented, for instance in mathematical, graphical, or in tabular form.

4.2 Uncertainties

The uncertainty to be stated as the uncertainty of the calibration of the temperature block calibrator is the measurement uncertainty with which the temperature in a boring of the calibrator can be stated.

This uncertainty is a component that must be used in the calculation of the uncertainty when a thermometer is calibrated against the temperature in a boring of the calibrator.

An example of the calculation of the measurement uncertainty is given in the Annex A.

The following contributions to the uncertainty of measurement shall be taken into account:

4.2.1 Deviation of the temperature shown by the indicator of the block calibrator from the temperature in the measurement zone

The contributions are essentially to be attributed to the calibration of the standard thermometer, the measurement performed with the standard thermometer, display resolution unit and differences between the measurements at decreasing and increasing temperature (hysteresis).

4.2.2 Temperature distribution in the measurement zone

Additional deviations of the indication of the built-in controlling thermometer from the temperature in the measurement zone are caused by the temperature distribution in the block, the loading of the block, and the stability with time. These additional deviations are assumed to be uncorrelated.

The contribution u_i to the measurement uncertainty is derived from the greatest temperature difference measured ($t_{max} - t_{min}$):

$$u_i^2\ (t) = (t_{max} - t_{min})^2/3$$

The contributions to the uncertainties according to sections 3.1 to 3.5 are to be linearly interpolated between the calibration points. Near room temperature, however, the contribution to the uncertainty in a temperature range which symmetrically extends around ambient temperature can be assumed to be constant.

Example:

Upon initial calibration of a temperature block calibrator in the temperature range -30 °C < t < +200 °C, carried out at an ambient temperature of 20 °C, the following is found as the greatest temperature differences in the homogeneous zone: 0.3 °C at t = -30 °C and 0.6 °C at t = +200 °C. In the temperature range of 20 °C ± 50 °C, i.e. from -30 °C to +70 °C, the greatest temperature difference occurring can be assumed to be 0.3 °C; in the temperature range from +70 °C to +200 °C, linear interpolation between 0.3 °C and 0.6 °C is to be carried out.

4.3 Uncertainty as a result of the temperature deviation due to heat conduction

Uncertainty contributions which are the result of temperature deviations due to heat conduction of thermometers shall be determined in all cases (that this arises both from the standard thermometer and an external controlling/customer reference thermometer).

5 REPORTING RESULTS

The calibration certificate in which the results of measurements are reported should be set out with due regard to the ease of assimilation by the user to avoid the possibility of misuse or misunderstanding. At least the deviation of the indication of the built-in controlling thermometer from the temperature in the measurement zone in the calibration together with their corresponding uncertainties and the description of the measurement zone should be reported.

It is recommended to enclose with each calibration certificate the "Recommendations of the EURAMET Technical Committee for Thermometry for the use of temperature block calibrators" (see Annex C).

The results of the investigations are to be documented in the calibration certificate.

ANNEX A Example of an uncertainty budget

Calibration of a temperature block calibrator at a temperature of 400 °C *(see warning in 1.1)*

The temperature t_S which has to be assigned to the identified measurement zone of the dry block calibration is determined by a calibrated measurement system (thermometer associated with its indicator)

The deviation from the temperature t_R read on the built-in temperature indicator:

$$\delta t = (t_R - t_S) + \delta t_S + \delta t_i + \delta t_H + \delta t_B + \delta t_L + \delta t_V$$

where the sources of correction and uncertainty are identified as follows:

δt_S	Standard thermometer uncertainty
δt_i	Resolution of the controlling thermometer
δt_H	Hysteresis in the increasing and decreasing branches of the measuring cycle.
δt_B	Inhomogeneity of temperature in the boring.
δt_L	Loading of the block with other thermometers.
δt_V	Temperature variations during the time of measurement.

This situation is chosen in order to indicate in the uncertainty budget an achievable uncertainty when calibrating the dry block. As indicated emphatically before, the uncertainty on the calibration of thermometers using a dry block will be usually much more larger in practice compared to the one presented in this example, because of the heat losses along the stem of the thermometer, that depends on the design of the sensor.

The following values are used in the example, and are for illustration only.

δt_S: Standard thermometer uncertainty

The standard thermometer uncertainty covers hysteresis, drift, non-linearity, selfheat, calibration and others.

It was estimated to be U = 0.03 °C (coverage factor k = 2)

NB: If the standard was calibrated in a liquid bath, the bias and uncertainties due to different "self heating" must be taken into account in the uncertainty budget.

***δti:* Resolution of the controlling thermometer.**

The controlling thermometer has a scale interval of 0.1 °C, giving temperature resolution limits of ±0.05 °C with which the temperature of the block can be uniquely set.

Note: If the indication of the built-in controlling thermometer is not given in units of temperature, the resolution limits shall be converted into equivalent temperature values by multiplying the indication with the relevant instrument constant.

δt_H Hysteresis effects.

The temperatures indicated show a deviation due to hysteresis in cycles of increasing and decreasing temperatures which is estimated to be within ±0.05 °C.

δt_B Inhomogeneity of temperature in the boring.

The deviations due to axial inhomogeneity of the temperature in the calibration boring have been estimated from readings for different immersion depths to be within 0.5 °C.

δt_L Block loading.

The influence of maximum loading on the temperature of the central hole was found to be 0.05 °C (arbitrary but realistic value, strongly dependent of cases).

δt_V Temperature stability

Temperature variations due to lack of temperature stability during the measuring cycle of 30 min are estimated to be within ±0.03 °C.

Uncertainty budget on the temperature deviation δt:

quantity x_i	Description	Estimate (°C)	Coverage Interval (°C)	Distribution	Coefficient	Uncertainty contribution (°C)
δt_S	Calibration of the standard	399.52				0.015
δt_i	Resolution of indicator	0.00	0.10	rectangular	2√3	0.029
δt_H	Hysteresis effects	0.00	0.05	rectangular	2√3	0.014
δt_B	Axial inhomogeneity	0.00	0.5	rectangular (*)	√3	0.289
δt_L	Loading effects	0.00	0.05	rectangular (*)	√3	0.029
δt_V	Stability in time	0.00	0.06	rectangular	2√3	0.017
δt		0.48				0.293 (**)

(*) asymmetric distribution

(**) which leads to an expanded (k = 2) uncertainty of: 0.6 °C

Reported result

The temperature to be assigned to the measurement zone when the temperature indicator shows 400 °C is 399.5 °C ± 0.6 °C.

The reported expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor k = 2.

ANNEX B Procedure for the determination of the influence of axial temperature distribution

Temperature block calibrators for the calibration of thermometers are usually used in different set-ups, sensing elements of different length being located in different areas of the measurement zone. As a result, the axial temperature distribution along the boring in the measurement zone makes a contribution to the uncertainty of calibration (which frequently dominates all other contributions). The determination of the axial temperature distribution is complicated because the thermometers themselves influence the temperature distribution. This influence can be complex, as, for example, a thermometer immersed to different depths leads to different heat conductions, which may, however, act on the transient behaviour of the block calibrator.

B.1.1 Determination of the temperature in three points using a sensor of short length

A thermometer with a maximum sensor length of 5 mm is used to determine the temperature at the lower end, in the middle and at the upper end of the measurement zone. The thermometer outside diameter should be ≤ 6 mm. In the temperature range from –100 °C to 250 °C, Pt resistance thermometers and in the range from 250 °C to 1300 °C thermocouples (including Pt/Pd thermocouples) are to be preferred.

Example: For a temperature block calibrator with a measurement zone 40 mm in length at the lower end of the boring, measurements under the following conditions are necessary:

(1) thermometer touching the lower end,

(2) raised/withdrawn 20 mm,

(3) raised 40 mm,

(4) thermometer touching the lower end.

B.1.2 Direct determination of temperature differences by means of a differential thermocouple

Here the temperature difference is directly measured using a differential thermocouple, the two junctions being about 25 mm apart. The differences can be measured at several points in the boring, from the lowest point (touching the lower end) upwards. The correct measurement of the temperature difference should be checked prior to the use of the differential thermocouples.

It is also possible to introduce two sheathed thermocouples with a small outside diameter together into the boring. While the first thermocouple remains at the lower end, the temperature differences from the second thermocouple are determined, which is at a known distance from the first thermocouple (for example, 20 mm and 40 mm). When both thermocouples are immersed to the same depth, an adjustment for the zero temperature difference is possible.

B1.3 Determination of the temperature at two points

If the temperature distribution is determined using a thermometer with a relatively long sensing element, shifting the thermometer by 40 mm (the usual length of the homogeneous zone of the block calibrator) will not be reasonable. Even so, for some calibrators a measurement at two different immersion depths (for example, touching the bottom and raised 20 mm) can furnish sufficient information about the influence of the temperature distribution on the contribution to the uncertainty of measurement.

It is to be noted that in accordance with section 4.2, the contribution to the uncertainty of measurement is determined in this case according to $u_i^2(t) = (t_1 - t_2)^2/3$.

ANNEX C Recommendations of the EURAMET TECHNICAL COMMITTEE "Thermometry" for the use of temperature block calibrators

Results reported in the calibration certificate have been obtained following the EURAMET Guideline cg-13. When the calibrator is used, the following points shall nevertheless be taken into consideration:

The calibration of temperature block calibrators mainly relates to the temperature of the block. The temperature of the thermometer to be calibrated in the block can deviate from this temperature. When a thermometer of the same type is used under measurement conditions identical to those during calibration, it can be assumed that the errors of measurement during the calibration of ideal thermometers are not greater than the uncertainties stated in the calibration certificate. If this is not the case (for instance use of different inserts or thermometers to those during calibration), the user of the block calibrator should confirm that the calibration results are still valid. Unless otherwise stated in the calibration certificate, it shall be ensured that

- the measuring element is in the measurement zone,
- the inside diameter of the boring used in the calibrator (and of the bushing, if present) is in the temperature range from -100 °C to +660 °C at most 0,5 mm, and in the temperature range from +660 °C to +1300 °C at most 1,0 mm, larger than the outside diameter of the thermometer to be calibrated. If this requirement can not be met, the customer must be aware that there will be an significant uncertainty contribution.

When thermometers are calibrated, an additional error of measurement due to heat conduction shall be taken into account. A good test for potential temperature deviations due to heat conduction is to check whether the display of the test thermometer changes when the thermometer is lifted up by 20 mm. Note that contributions to the uncertainty of measurement due to the thermometer to be calibrated (e.g. inhomogeneities of thermocouples) are not included in the measurement uncertainty of the calibrator.

The data given in the calibration certificate are decisive for the calibration, not the manufacturer's specifications. Before starting calibration, please discuss by all means the calibration and operating conditions with your calibration laboratory.

In all cases, the user must provide himself the means to control the metrological quality of the instrument.