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Calibration of Temperature Block Calibrators

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Calibration Guide

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CALIBRATION OF TEMPERATURE BLOCK CALIBRATORS

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Purpose

This document has been produced to improve harmonisation in calibration of temperature block calibrators. It gives advice to calibration laboratories to establish practical procedures and the calculation of uncertainties.

Authorship

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Contents

Section **Page**

Contents 3

1 SCOPE 4

2 CALIBRATION CAPABILITY 4

3 CALIBRATION 5

4 UNCERTAINTY OF MEASUREMENT 7

5 REPORTING RESULTS 9

ANNEX A Example of an uncertainty budget 10

ANNEX B Procedure for the determination of the influence of axial temperature distribution 14

ANNEX C Recommendations of the EURAMET TECHNICAL COMMITTEE "Thermometry" for the use of temperature block calibrators 16

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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 in the borings of this block. A temperature block calibrator comprises at least the solid-state block, a temperature-regulating device for the block, a temperature sensor with indicator (the built-in controlling thermometer) to determine the block temperature. These components are either combined to form a compact unit, or an unambiguous assignment of these components to each other shall be possible.

1.2 This Guideline is valid in the temperature range from -80 °C to +1300 °C. The temperature ranges stated by the manufacturer shall not be exceeded.

1.3 EA Publication EA-10/08 (previously EAL-G31) should be applied if appropriate.

2 CALIBRATION CAPABILITY

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

2.2 The temperature sensor and indicator used to determine block temperature shall meet the requirements which would be necessary, if they were calibrated separately from the block.

2.3 The borings used for calibrations shall have a zone of sufficient temperature homogeneity of at least 40 mm in length (in the following referred to as measurement zone), whose position is exactly specified. The homogeneous zone will in general be at the lower end of the boring. If the homogeneous zone is situated at another place, this shall explicitly be stated.

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

2.4.1 In the temperature range from –80 °C to +660 °C, the inside diameter of the boring or bushing used 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.

The immersion depth of the thermometer shall at least be equal to fifteen times the outer diameter of the thermometer. Some thermometer constructions may require a larger immersion depth.

3 CALIBRATION

3.0.1 For the purpose of calibration, it is assumed that any required adjustments have been performed before the calibration is started.

3.0.2 When a temperature block calibrator is calibrated, the special characteristics of the temperature distribution in the block of the calibrator (defined in sections 3.1 to 3.5) are to be investigated and documented, in addition to the deviation of the temperature in the homogeneous zone from the temperature indicator of the calibrator. If previous investigations with calibrators of the same type are used for the determination of the characteristics or uncertainties of measurement, the investigation reports available must be referred to in the calibration certificate.

3.0.3 All investigations shall be carried out under the measurement conditions stated in section 2.3.

3.0.4 If adapter bushings are required to comply with the requirement of section 2.4.1, these shall be made of the material proposed by the manufacturer.

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

3.0.6 The thermometer used for the investigations according to sections 3.1 to 3.4 (test thermometer) need not be calibrated, as these tests are performed to measure the temperature differences (exception: measurements under point 4 in Annex A). The sensitivity at the measuring temperature shall, however, be known with sufficiently small measurement 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 shall be tested.

3.0.7 Unless otherwise agreed with the client, the following measurement conditions are to be complied with:

- All measurements are to be carried out with thermometers with an outside diameter $d \leq 6$ mm.
- All measurements, with the exception of those mentioned in section 3.1, are to be carried out in such a way that the thermometer touches the lower end of the boring.

3.0.8 The following investigations are to be carried out in particular:

3.1 Axial temperature homogeneity along the boring in the measurement zone

3.1.1 The influence of the temperature distribution in the measurement zone along the boring (axial temperature distribution) is to be determined in such a way that it can be taken into account in the uncertainty of measurement of the calibration. Potential methods are represented in Annex B. Previous investigations with calibrators of the same type can be used for the determination of the contribution to the uncertainty of measurement. It is possible in agreement with the client to investigate only the influence of the axial temperature distribution on certain types of thermometers.

The measurement is to be performed in the central boring or in a boring particularly marked.

The necessary investigations are to be carried out at the operating temperature showing the greatest difference from the ambient temperature. For temperature block calibrators whose measurement zone can be both heated and cooled, the investigations are to be carried out at the highest and at the lowest operating temperature. The influence of the temperature distribution at other operating temperatures can be estimated by linear interpolation (cf. example in section 4.2).

3.2 Temperature differences between the borings

3.2.1 The greatest temperature difference occurring between the borings 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 are determined. The measurement of the temperature difference between (opposite) borings situated at as great a distance from each other as possible is of particular importance.

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

In the case of very small uncertainties of measurement, more detailed investigations into the influence on the temperature in the measurement zone due to different loading are necessary. It is recommended the compare the results for loading with only one thermometer and all borings loaded. Loadings with thermometers can be simulated by loadings with metal or ceramic bars. The measurements are to be carried out at the temperature with the largest temperature difference to room temperature.

3.4 Stability with time

3.4.1 The maximum range of temperatures indicated by a sensor in the measurement zone over a 30 minute period, when the system has reached equilibrium, shall be determined.

Measurements are to be performed at three different test temperatures: at the highest test temperature, at the lowest test temperature and at room temperature. If the highest or lowest test temperature corresponds to room temperature, the third test temperature shall be selected in the middle of the temperature range tested.

3.5 Temperature deviation due to heat conduction

3.5.1 In agreement with the client, the temperature error due to heat conduction is to be determined for such thermometers which are to be calibrated at the client's. This deviation is not part of the temperature block calibrator's measurement uncertainty, but is to be taken into account separately when the temperature block calibrator is used. Temperature deviations due to heat conduction need not be taken into account for thermometers with outside diameters of $d \leq 6$ mm.

3.6 Determination of the deviation of the indication of the built-in controlling thermometer from the temperature in the measurement zone

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

3.6.1 Measurements

3.6.1.1 The determination of the deviation of the temperature given by the indicator of the block calibrator from the temperature in the measurement zone is performed in the central boring or in a particularly marked boring. Measurement at a minimum of three different temperatures (calibration points) are to be carried out, which are distributed as uniformly as possible over the required temperature range. At each calibration point two measurement series are carried out, in which for a period of at least 10 minutes 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 at the other at decreasing temperatures. Results obtained in tests carried out to determine the stability with time may be used without repeat measurement, provided a calibrated thermometer had been used. Measurements at increasing and decreasing temperatures are not required for the highest and the lowest calibration point if the temperature coincides with the highest or lowest operating temperatures specified by the manufacturer. However, at least two measurement series are to be recorded, between which the operating temperature of the calibrator was changed.

3.6.2 Evaluation

3.6.2.1 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 in mathematical, graphical, or in tabular form.

4 UNCERTAINTY OF MEASUREMENT

4.0.1 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. If the temperature deviation due to heat conduction may be neglected, this measurement uncertainty is to be equated with the measurement uncertainty a user can expect for a thermometer when he calibrates this thermometer with the temperature block calibrator and conscientiously complies with the operating instructions and the provisions in this Calibration Guideline.

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

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

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

4.1.1 The contributions are essentially to be attributed to the calibration of the standard thermometer, the measurement performed with the standard thermometer, the resolution of the digital display unit and differences between the measurements at decreasing and increasing temperature (hysteresis). The measurement uncertainties are determined by analogy with the procedure used for the calibration of a thermometer.

4.2 Temperature distribution in the block

4.2.1 Additional deviations of the indication of the built-in controlling thermometer from the temperature in the measurement zone used by the client (which might be different from the zone used for the measurements described in 3.6) are caused by the not exactly known temperature distribution in the block, the loading of the block, and the stability with time. These additional deviations are not correlated. The resulting contributions to the measurement uncertainty can be estimated from the measurements according to 3.1 to 3.4.

The contribution u_i to the measurement uncertainty is derived from the greatest temperature difference measured $(t_{max} - t_{min})$: $u_i^2\ (t) = (t_{max} - t_{min})^2/12$ if at least three individual measurements were carried out. If the contribution u_i to the uncertainty of measurement is determined from only two individual measurements, the following relation is to be used: $u_i^2\ (t) = (t_1 - t_2)^2/3$.

4.2.2 The contributions to the uncertainties according to sections 3.1 to 3.4 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

4.3.1 Uncertainty contributions which are the result of temperature deviations due to heat conduction of thermometers with outside diameters $d \leq 6$ mm can be neglected. If thermometers with $d > 6$ mm are used, this contribution to uncertainty shall be separately analysed.

5 REPORTING RESULTS

5.1 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's mind to avoid the possibility of misuse or misunderstanding.

5.2 The certificate shall meet the requirements of EA Publication EA-4/01 (previously EAL-R1).

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

5.4 The results of the investigations carried out under points 3.1 to 3.4 are to be documented in the calibration certificate.

ANNEX A Example of an uncertainty budget[1]

A1 Calibration of a temperature block calibrator at a temperature of 180 °C

A.1.1 The temperature which has to be assigned to the temperature sensing area of a thermometer inserted into one of the calibration borings of temperature block calibrator with a built-in temperature indicator is determined by comparison with a calibrated platinum resistance thermometer as a reference standard at 180 °C. The temperature indicated by the reference standard is determined by a measurement of its electrical resistance in an ac resistance bridge.

A.1.2 The temperature t_X, that has to be assigned as the temperature of the boring when the reading of the built-in temperature indicator is 180 °C is given by:

$$t_X = t_S - \delta t_S + \delta t_D - \delta t_i + \delta t_R + \delta t_H + \delta t_B + \delta t_L + \delta t_V \qquad \text{(A1)}$$

where:

t_S	temperature of the reference thermometer derived from the ac resistance measurement;
δt_S	temperature correction due to the ac resistance measurement;
δt_D	temperature correction due to drift in the value of the reference standard since its last calibration;
δt_i	temperature correction due to limited resolution of the built-in temperature indicator;
δt_R	temperature difference between borings;
δt_H	temperature correction due to hysteresis in the increasing and decreasing branches of the measuring cycle;
δt_B	temperature correction due to axial inhomogeneity of temperature in the borings;
δt_L	temperature correction due to differences in the loading of the block with thermometers to be calibrated; and
δt_V	temperature variations during the time of measurement.

A.1.3 Temperature corrections due to stem conduction are not considered; the platinum resistance thermometer used as reference has an outer diameter $d \leq 6$ mm. Prior investigations have shown that stem conduction effects can be neglected in this case.

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[1] A similar example will be found in EA Publication EA-4/02-S2 where information is given on the differences between the two expamles.

A.1.4 **Reference standards (t_S):** The calibration certificate of the resistance thermometer used as reference standard states for the measured temperature value 180,10 °C the expanded uncertainty of measurement U = 30 mK (coverage factor k = 2).

A.1.5 **Determination of the temperature by resistance measurement(δt_S):** The temperature of the resistance thermometer used as reference standard is determined as 180,10 °C. The standard uncertainty associated with the electrical measurement converted to temperature corresponds to $u(\delta t_S) = 10$ mK .

A.1.6 **Drift of the temperature of the reference standard (δt_D):** From general experience with platinum resistance thermometers of the type used as reference standard in the measurement, the change of the temperature due to resistance ageing is estimated to be within the limits of ±40 mK.

A.1.7 **Resolution of the built-in controlling thermometer (δt_i):** The built-in controlling thermometer has a scale interval of 0,1 K, giving temperature resolution limits of ± 50 mK with which the thermodynamic state of the temperature 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.

A.1.8 **Temperature difference between borings (δt_R):** The calibrators has 6 holes. The largest temperature difference measured at 180 °C between the holes was 140 mK, leading to an assumed temperature distribution between the holes with limits of ± 70 mK

A.1.9 **Hysteresis effects (δt_H):** The temperatures indicated show a deviation due to hysteresis in cycles of increasing and decreasing temperatures which is estimated to be within ± 50 mK.

A.1.10 **Axial inhomogeneity of temperature (δt_B):** 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 ± 250 mK.

A.1.11 **Block loading (δt_L):** The influence of maximum loading on the temperature of the central hole was found to be 50 mK. (See 3.3)

A.1.12 **Temperature instability (δt_V):** Temperature variations due to temperature instability during the measuring cycle of 30 min are estimated to be within ± 30 mK.

A.1.13 **Correlations:** None of the input quantities are considered to be correlated in this model.

A.1.14 **Repeated observations:** Due to the finite resolution of the indication of the built-in thermometer, no scatter in the indicated values has been observed.

A.1.15 **Uncertainty budget:**

quantity X_i	**Description**	**Estimate** x_i	**Standard uncertainty** $u(x_i)$	**Distribution**	**Sensitivity coefficient** c_i	**Uncertainty contribution** $u_i(y)$
t_S	Temp. of reference thermometer	180,10 °C	15 mK	normal	1,0	15 mK
δt_S	Corr. for resistance measurement	0,00 K	10 mK	normal	1,0	-10 mK
δt_D	Drift of resistance thermometer	0,00 K	23 mK	rectangular	1,0	23 mK
δt_{iX}	Resolution of indicator	0,00 K	29 mK	rectangular	1,0	-29 mK
δt_R	Temp. diff. between borings	0,00 K	40 mK	rectangular	1,0	40 mK
δt_H	Hysteresis effects	0,00 K	29 mK	rectangular	1,0	29 mK
δt_B	Axial inhomogeneity	0,00 K	144 mK	rectangular	1,0	144 mK
δt_L	Loading effects	0,00 K	29 mK	rectangular	1,0	29 mK
δt_V	Stability in time	0,00 K	17 mK	rectangular	1,0	17 mK
t_X		180,10 °C				161 mK

A.1.16 **Expanded uncertainty**

The standard uncertainty of measurement associated with the result is dominated by the effect of the unknown temperature correction for the axial temperature inhomogeneity in the measurement boring and (to a smaller extent) by the radial temperature difference between the measurement borings. The resulting distribution is not a normal distribution but essentially trapezoidal. For details,

cf. EA-4/02-E2 section S10.13. For the example above an edge parameter β = 0,563 is obtained, which for a coverage probability of 95% leads to a coverage factor k = 1,74.

$U = ku(t_X)$ = 1,74 . 0,161 K ≅ 0,3 K

A.1.17 **Reported result**

The temperature to be assigned to the temperature sensing area of a thermometer inserted in one of the calibration borings when the built-in temperature indicator shows 180 °C is 180,10 °C ± 0,32 °C.

The reported expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor $k = 2$, which for a normal distribution corresponds to a coverage probability of approximately 95 %.

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, sensors 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 difficult, as a test thermometer itself influences this 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.

This is why a procedure for the determination of the influence of the axial temperature distribution is to be selected which meets the clients' requirements as closely as possible. Such procedures may, for example, be:

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 may be provided with a protective tube with an outside diameter $d \leq 6$ mm. In the temperature range from –80°C to 250°C, Pt resistance thermometers and in the range from 250°C to 1300°C thermocouples (also Pt-Pd thermocouple) are to be preferably used.

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) pulled out 20 cm,

(3) pulled out 40 cm,

(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 in one or several points in the boring to the lowest point of the boring (touching the lower end) using a differential thermocouple.

For this purpose, a completely conditioned thermocouple can, for example, be used, the measurement points being spaced about 25 mm. In a bath or in a heat pipe it should be checked at regular intervals whether the temperature difference of 0 K is correctly measured.

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). If both thermocouples are immersed to the same depth, an adjustment for the temperature difference 0 K is possible.

B1.3 Determination of the temperature in two points

If the temperature distribution is determined with the aid of a thermometer with a relatively long sensor, shifting of the thermometer by 40 mm (the usual length of the homogeneous zone of the block calibrator) will not be reasonable. It has turned out for some calibrators that a measurement at two different immersion depths (for example, striking the bottom and pulled out 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 $u_i^2(t) = (t_1 - t_2)^2/3$.

B1.4 Determination of the temperature with calibrated thermometers with different sensor length

If information about which thermometers are calibrated in the block calibrator is available, the influences of the axial temperature distribution on the thermometer type can be determined directly. For this purpose, measurements with the individual thermometer types have to be carried out. If information about the thermometers to be calibrated is not available, the measurement should be performed with two thermometers of different type, if possible.

It is to be noted that for these measurements as stated under point 4 all thermometers used must have been calibrated.

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

C1.1 Results reported in the calibration certificate have been obtained following the EA Guidelines EA-10/x3. When the calibrator is used, the following points shall nevertheless be taken into consideration:

C1.2 The calibration of temperature block calibrators mainly relates to the temperature of the solid-state 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. Unless otherwise stated in the calibration certificate, it shall be ensured that

- the measuring element is in the homogeneous temperature zone;
- the inside diameter of the boring used in the calibrator (possibly of the bushing) is in the temperature range from -80 °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;
- the immersion depth of the thermometer to be calibrated is at least equal to 15 times the outside diameter of the thermometer to be calibrated; and
- the thermometer to be calibrated has a diameter of $d \leq 6$ mm.

C1.3 Please check in particular whether a heat-conveying means (for instance oil) was used for the calibration of your temperature block calibrator. If so, the calibration is valid only if the calibrator is used with a corresponding heat-conveying means.

C1.4 When thermometers with outside diameters of $d > 6$ mm are calibrated, an additional error of measurement due to heat conduction shall be taken into account. If such measurements are to be carried out, your calibration laboratory can determine the additional heat conduction for the thermometer type investigated by you. 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. Contributions to the uncertainty of measurement due to the thermometer to be calibrated by you (e.g. inhomogeneities of thermocouples) are not included in the measurement uncertainty of the calibrator either.

C1.5 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.

C1.6 Unless otherwise stated in the calibration certificate, it shall be ensured (independent of the manufacturer's specifications) that

- the calibrator is operated in the vertical position;
- no additional thermal insulation is used; and
- the environmental temperature is (23 ± 5) °C.

C1.7 To check the temperature block calibrator it is recommended to carry out check measurements at regular intervals using a calibrated thermometer. If such check measurements with a calibrated thermometer are not made, it is urgently recommended to recalibrate the temperature block calibrator annually.