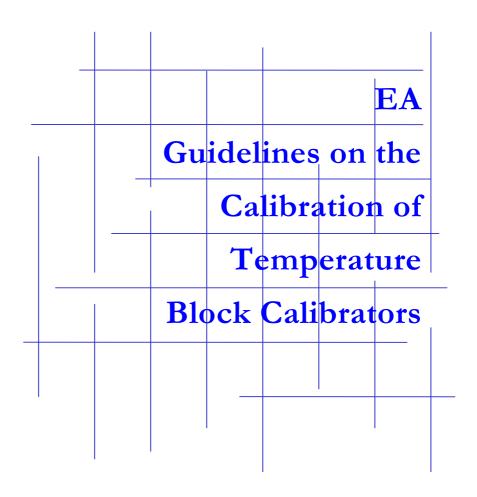


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PURPOSE

This document been produced by EA to improve the harmonisation in the calibration of temperature block calibrators. It provides guidance to national accreditation bodies to set up minimum requirements for the calibration of temperature block calibrators and gives advice to calibration laboratories to establish practical procedures and the evaluation of uncertainties. This document was approved by the EA General Assembly in November 1999.

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Authorship

This document has been prepared by EA Committee 2 (Technical Activities), based on a draft produced by the EA Expert Group "Temperature and Humidity".

Official language

The text may be translated into other languages as required. The English language version remains the definitive version.

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Guidance Publications

This document represents a consensus of EA member opinion and preferred practice on how the relevant clauses of the accreditation standards might be applied in the context of the subject matter of this document. The approaches taken are not mandatory and are for the guidance of accreditation bodies and their client laboratories. Nevertheless, the document has been produced as a means of promoting a consistent approach to laboratory accreditation amongst EA member bodies, particularly those participating in the EA Multilateral Agreement.

Further information

For further information about this publication, contact your national member of EA. Please check our website for up-to-date information http://european-accreditation.org

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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.
- 2.4.2 Any insulation materials recommended by the manufacturer to be placed on top of the block shall be defined (e.g. material and thickness) and supplied with the apparatus. The insulation shall be used in the calibration of the block and in its subsequent use.

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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.
- 3.0.3 All investigations shall be carried out under the measurement conditions stated in section 2.3. Exception: When the homogeneity of the temperature in the measurement zone is investigated, it shall be ensured only at the maximum immersion depth that the immersion depth is at least equal to 15 times the outer diameter of the thermometer.
- 3.0.4 If adapter bushings are required to comply with the requirement of section 2.3.1, 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. 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 As far as calibration is concerned, a distinction is made between initial and repeat calibration. The initial calibration is the first calibration of the temperature block calibrator by an accredited laboratory. A repeat calibration (the second or any further calibration) of the temperature bloc calibrator can be carried out if the calibration certificate of the initial calibration, comprising all important data, is available. If the results of the repeat calibration differ from the initial calibration by more than the reported uncertainty of the measurement, then the initial calibration shall be repeated and consideration given to increasing the uncertainty to be stated on the certificate.
- 3.0.8 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 ≤ 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.
 - All measurements are to be carried out with the top of the block exposed, or insulated, as recommended by the manufacturer.

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3.0.9 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 greatest temperature difference occurring in the measurement zone is to be determined. For this purpose, the temperature is determined at the lower end, in the middle and at the upper end of the measurement zone, using a thermometer with a sensor length not exceeding 5 mm. The thermometer may be provided with a protective tube (outside diameter: $d \le 6$ mm). It is recommended to use Pt resistance thermometers in the temperature range from -80 °C to +660 °C and noble metal thermocouples (including Pt-Pd thermocouples) in the temperature range from +660 °C to +1300 °C. Base metal thermocouples are unsuitable for these measurements.

Example: The following measurements are required for a temperature block calibrator with a measurement zone 40 mm in length at the lower end of the boring:

- 1) thermometer touching the ground;
- 2) thermometer pulled out 20 mm;
- 3) thermometer pulled out 40 mm; and
- 4) thermometer touching the ground.
- 3.1.2 The measurement is to be performed in the central boring or in a particularly marked boring.
- 3.1.3 **Initial calibration:** Measurements are to be carried out at the highest and at the lowest temperature of the measuring range. If one of these measurement points is at room temperature, the temperature for this measurement point is to be increased or decreased by 20 °C.
- 3.1.4 **Repeat calibration:** Measurements are to be performed at that temperature of the measurement range whose difference to room temperature is greatest.

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.
- 3.2.2 **Initial calibration:** The temperatures are determined in at least three borings distributed as uniformly as possible on the greatest reference circle of the temperature block. If the calibrator is provided with less than 4 borings, the temperature differences are determined by measurements in the borings and cyclic exchange of the thermometers, or they are determined directly with calibrated thermometers.
- 3.2.3 **Repeat calibration:** The temperature difference is determined between those two borings which had shown the greatest temperature difference upon initial calibration.

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

- 3.3.1 **Initial calibration:** These measurements are carried out to determine the change of the temperature difference between the reference thermometer and a test thermometer, which occurs when further borings are loaded with thermometers or suitable sheaths. The sheaths or thermometers shall protrude from the respective boring by at least 200 mm. Maximum possible loading with thermometers/bushings of 6 mm or smaller in diameter shall be ensured. The measurements are to be carried out at the temperature of the measurement range which shows the greatest temperature difference with respect to room temperature.
- 3.3.2 **Repeat calibration:** A repeat measurement is not required.

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.
- 3.4.2 Initial calibration and repeat calibration: 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 $d \le 6$ 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. The same measurements are to be performed for initial and repeat calibration.
- 3.6.0.2 In the case of temperature block calibrators where the built-in controlling thermometer is introduced into a boring from which it can be removed, calibration of this thermometer according to a different calibration guideline is recommended.

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 ad-

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justment of the temperature at 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 contributions u_i to the uncertainty of measurement are derived from the greatest temperature difference ($t_{\text{max}} - t_{\text{min}}$) measured:

$$u_i^2(t) = (t_{\text{max}} - t_{\text{min}})^2 / 12.$$

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- 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 $-30 \,^{\circ}\text{C} < t < +200 \,^{\circ}\text{C}$, carried out at an ambient temperature of 20 $\,^{\circ}\text{C}$, the following is found as the greatest temperature differences in the homogeneous zone: 0,3 $\,^{\circ}\text{C}$ at $t = -30 \,^{\circ}\text{C}$ and 0,6 $\,^{\circ}\text{C}$ at $t = +200 \,^{\circ}\text{C}$. In the temperature range of 20 $\,^{\circ}\text{C} + 50 \,^{\circ}\text{C}$, i.e. from $-30 \,^{\circ}\text{C} + 70 \,^{\circ}\text{C}$, the greatest temperature difference occurring can be assumed to be 0,3 $\,^{\circ}\text{C}$; in the temperature range from $+70 \,^{\circ}\text{C}$ to $+200 \,^{\circ}\text{C}$, linear interpolation between 0,3 $\,^{\circ}\text{C}$ and 0,6 $\,^{\circ}\text{C}$ 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 $d \le 6$ 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 EA Expert Group 'Temperature and Humidity' for use of temperature block calibrators" (see Annex B).
- 5.4 The results of the investigations carried out under points 3.1 to 3.4 are to be documented in the calibration certificate.

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ANNEX A EXAMPLE OF AN UNCERTAINTY BUDGET¹

A.1 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:

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

ts - temperature of the reference thermometer derived from the ac resistance measurement;

 $\delta t_{\rm S}$ - 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;

 $\delta t_{\rm R}$ - temperature difference between borings;

 δt_{H} - temperature correction due to hysteresis in the increasing and decreasing branches of the measuring cycle;

 $\delta t_{\rm B}$ - temperature correction due to axial inhomogeneity of temperature in the borings;

 $\delta t_{\rm L}$ - 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 $d \le 6$ mm. Prior investigations have shown that stem conduction effects can be neglected in this case.
- 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(\delta t_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 $u(\delta t_s) = 10 \text{ mK}$.
- A.1.6 **Drift of the temperature of the reference standard (\delta t_D):** From general experience with platinum resistance thermometers of the type used as reference standard in the

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

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 \pm 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 (\delta t_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 (\delta t_L):** The influence of maximum loading on the temperature of the central hole was found to be 50 mK.
- A.1.12 **Temperature instability (\delta t_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	estimate	standard uncertainty ²	probability distribution	Sensitivity coefficient	uncertainty contribution
X_{i}	X_i	$u(x_i)$		$c_{_i}$	$u_i(y)$
$t_{ m S}$	180,10 °C	15 mK	normal	1,0	15 mK
δt_{S}	0,0 K	10 mK	normal	-1,0	-10 mK
δt_{D}	0,0 K	23 mK	rectangular	1,0	23 mK
δt_{iX}	0,0 K	29 mK	rectangular	-1,0	-29 mK
δt_{R}	0,0 K	40 mK	rectangular	1,0	40 mK
δt_{H}	0,0 K	29 mK	rectangular	1,0	29 mK
δt_{B}	0,0 K	144 mK	rectangular	1,0	144 mK
δt_{L}	0,0 K	29 mK	rectangular	1,0	29 mK
δt_{V}	0,0 K	17 mK	rectangular	1,0	17 mK
t_{X}	180,10 °C				161 mK

The figures given below were derived on the basis of the information on the various uncertainty components as given above according to EA Publication EA-4/02 assuming the indicated probability distribution.

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A.1.16 Expanded uncertainty

The expanded uncertainty associated with the measurement of the temperature of the furnace is

$$U = k u(t_x) = 2.161 \text{ mK} \approx 0.32 \text{ K}$$

Note: The main contribution to the uncertainty is the axial inhomogeneity of temperature (δt_L) with a rectangular distribution. Therefore a coverage factor of k = 2 probably leads to a coverage probability of more than 95 %.

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 $180 \,^{\circ}\text{C}$ is $180,10 \,^{\circ}\text{C} \pm 0,32 \,^{\circ}\text{C}$.

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 %.

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ANNEX B RECOMMENDATIONS OF THE EA EXPERT GROUP "TEMPERATURE AND HUMIDITY" FOR THE USE OF TEMPERATURE BLOCK CALIBRATORS

- B1.1 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:
- B1.2 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 $d \le 6$ mm.
- B1.3 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.
- B1.4 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.
- B1.5 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.
- B1.6 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.
- B1.7 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.

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