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Comparison Report DKD-V 5.1

Calibration of temperature block calibrators at temperatures above 600 °C / Pilot study to determine the measurement uncertainty

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Deutscher Kalibrierdienst (DKD) – German Calibration Service

Since its foundation in 1977, the DKD brought together calibration laboratories of industrial enterprises, research institutes, technical authorities, inspection and testing institutes. On 3 May 2011, the DKD was reestablished as a *technical body* of the PTB and the accredited laboratories.

This body is called *Deutscher Kalibrierdienst* (DKD – *German Calibration Service*) and is under the direction of the PTB. The guidelines and guides elaborated by the DKD represent the state of the art in the respective technical areas of expertise and can be used by the *Deutsche Akkreditierungsstelle GmbH* (the German accreditation body – DAkkS) for the accreditation of calibration laboratories.

The accredited calibration laboratories are now accredited and monitored by the DAkkS as legal successor of the DKD. They carry out calibrations of measuring devices and measuring standards for the measured values and measuring ranges defined during accreditation. The calibration certificates issued by these laboratories prove the traceability to national standards as required by the family of standards DIN EN ISO 9000 and DIN EN ISO/IEC 17025.

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Foreword

The DKD comparison reports aim to disclose the results of comparison measurements organized, carried out or evaluated by the German Calibration Service. They contain a lot of information regarding the measurement capabilities of the participating calibration laboratories and the comparability of the measurements. The DKD comparison reports reflect the views of the respective authors which do not necessarily represent in detail the perspective of the Board of the DKD or that of the Technical Committees.

The DKD comparison reports are aimed at presenting the examined aspects and results of the calibration and shall be made available, both nationally and internationally, to the big community of calibration laboratories through publication by the DKD.

The DKD comparison reports are intended to depict the aspects considered during the interlaboratory comparison as well as the results of the calibration. Through the publication by the DKD, theses reports shall be made available to the large community of calibration laboratories, both nationally and internationally.

The expert and comparison reports do not have to be approved by the Executive Board (resolution made at the 5th DKD Executive Board meeting), but only by the respective Technical Committee and the Executive Office of the DKD.

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1 Introduction and objectives

The purpose of the interlaboratory comparison / pilot study was to examine the behaviour of temperature block calibrators in the temperature range above 600 °C.

So far, the estimation of the measurement uncertainties for the calibration of block calibrators at high temperatures was not based on real measurements.

Investigations carried out by the PTB in 2011 have shown that this estimation of the measurement uncertainties does not properly reflect the real influences.

It was therefore considered necessary to determine the influences within an interlaboratory comparison / pilot study and to agree on a procedure for calculating the measurement uncertainty.

Based on the results determined within the scope of the comparison, the aim was to quantify the influences on the calibration result and to derive the smallest achievable uncertainties for the calibration of block calibrators at temperatures above 600 °C.

The investigation of the axial homogeneity and accordingly the heat dissipation were given special attention, since these influences represent a very large contribution to the measurement uncertainty. Various types of thermometers and thermocouples were used to determine the influence as accurately as possible.

Paragraph 5 specifies the standard measuring equipment used by the individual participants.

The interlaboratory comparison / pilot study was carried out in coordination with PTB's Working Group 7.42 in Berlin. The calibration laboratory D-K-15186-01 of the ZMK & ANALYTIK GmbH assumed the function of pilot laboratory.

In a first step, the participants and the pilot laboratory submitted their measurement results to Working Group 7.42 at PTB. Subsequently, all measurement results were transferred to the pilot laboratory for evaluation.

Two temperature block calibrators were provided by two manufacturers.

One of the two block calibrators did not meet the stability requirements for comparative measurement objects and was therefore not considered in the final evaluation.

The participants of the interlaboratory comparison worked according to their accredited calibration procedures which are based on the guideline DAkkS-DKD-R 5-4 [1].

2 Participants

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3 Time frame of the interlaboratory comparison / pilot study

The pilot study was organised in the form of an interlaboratory comparison.

The PTB and the pilot laboratory carried out a calibration of the temperature block calibrator at the beginning and at the end of the interlaboratory comparison to ensure the measurement result and to verify the stability of the calibration item.

Halfway through the interlaboratory comparison, the first participants submitted their measurement results to PTB to be checked for plausibility. After a positive check by PTB, the interlaboratory comparison was continued.

The task of the pilot laboratory was to accompany the process and evaluate the measurement results in coordination with PTB. The methodical approach complied with the standard ISO/IEC 17043:2010 [2].

The course of the interlaboratory comparison / pilot study is shown in Fig. 1.

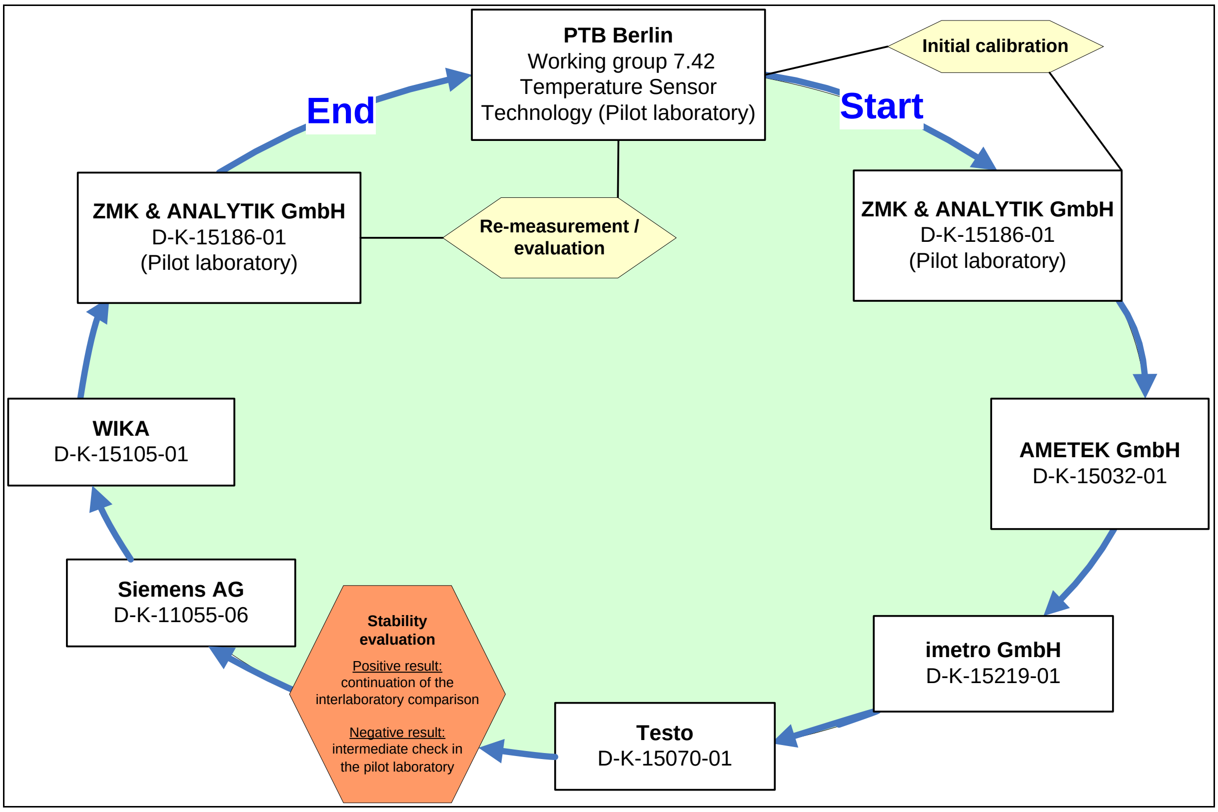


Fig. 1: Course of the interlaboratory comparison / pilot study V/0005/13 "Calibration of temperature block calibrators at temperatures above 600 °C / Pilot study to determine the measurement uncertainty"

The calibration items were made available to the participants for an agreed period. The calibration results were to be reported no later than two weeks after completion of the measurements. Occasionally, there were some delays. These delays were coordinated with the pilot laboratory and the participants which were next in line. In case of one participant, there was a considerable delay in submitting the measurement results. In agreement with PTB, this delay was tolerated.

The periods over which the measurements of the individual participants took place are shown in Table 1 below.

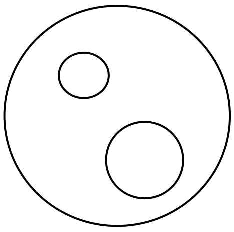
Table 1: Periods of measurement of the individual participants

Period	Activity
05/2012 to 09/2012	Planning phase
10/2012 - 02/2013	Initial calibration at PTB
04/2013	Initial calibration in the pilot laboratory (ZMK)
14.05. to 26.06.2013	Calibration in D-K-15032-01 (Ametek)
27.06. to 12.08.2013	Calibration in D-K-15219-01 (imetrologie)
13.08. to 02.09.2013	Calibration in D-K-15070-01 (testo)
29.08.2013	Intermediate evaluation by PTB: Evaluation of the stability of the calibration item based on the previous measurement results of the participants
03.09. to 26.09.2013	Calibration in D-K-11055-06 (Siemens)
27.09. to 21.10.2013	Calibration in D-K-15105-01 (WIKA)
22.10.2013 to 05.01.2014	Final calibration in the pilot laboratory (ZMK)
07.01. to 10.04.2014	Final calibration at PTB
05/2014 to 05/2015	Evaluation and assessment (pilot laboratory and PTB)
03/2015	Submission of the last results of a participant
05.06.2015	Presentation of the results (anonymised) at the meeting of the DKD Technical Committee “Temperature and Humidity”
06/2015 to 04/2016	Coordination with PTB / Preparation of the draft report

4 Subject of the interlaboratory comparison / pilot study

The following temperature block calibrator was provided by Ametek GmbH.

Temperature block calibrator



Type:	CTC-1200A
Manufacturer:	Ametek
Serial No.:	603307-00615
Measurement range:	300 °C to 1205 °C
Display resolution:	0.1 K
Insertion tubes:	Material: metal
	Insert 1: holes: 5.1 mm // 7.5 mm hole (obligation)
	Insert 2: holes: 5.1 mm // 6.5 mm hole (optional)
	Immersion depth: 102 mm (136 mm with insulation plug)
Accessories:	6 x ceramic plugs
	power plug
	tube tongs
	various connecting cables
	thermal protection shield
	various operator documentations

Stability of the calibration item

The stability of the calibration item was checked by the initial calibration and the repeated measurements at PTB and ZMK. In addition, a stability analysis was performed in the middle of the interlaboratory comparison. To this end, the first participants had previously sent their measurement results obtained at 1200 °C to PTB.

The results were checked for plausibility and evaluated. Based on this examination, it was determined that there was no need for an intermediate inspection of the block calibrator at the pilot laboratory, and so the interlaboratory comparison continued at the laboratory of the next participant.

Table 2 shows the results of the initial calibrations and the calibration at the end of the interlaboratory comparison carried out by ZMK.

Table 2: Results of the stability control of the calibration item in the pilot laboratory D-K-15186-01 of the ZMK & ANALYTIK GmbH

Measuring tempera-ture in °C	**Initial calibration** *Detected deviation of the calibration item*			**Calibration at the end of the interlaboratory comparison** *Detected deviation of the calibration item*	Expanded measurement uncertainty (k = 2) in K
	Deviation Measure-ment 1 in K	Deviation Measure-ment 2 in K	Deviation Measure-ment 3 in K	Deviation in K	
Insert 1 // Hole diameter 7.5 mm					
600	2.0	1.9	1.9	2.0	2.4
960	2.8	2.8	2.5	2.6	7.0
1200	2.3	2.1	2.1	2.3	9.2

The results of the stability investigations show a good agreement within the scope of the expanded measurement uncertainty. Thus, the required stability of the calibration item over the period in which the of the interlaboratory comparison was carried out has been confirmed.

Inbound inspection by the participants / Transport of the calibration items

The calibration item and the accessories were checked by the participants for completeness and integrity upon receipt.

The pilot laboratory was informed about the receipt and the result of the inbound inspection.

A carrying case for the calibration item had been provided by the manufacturer.

The transport to the next participant was carried out by an employee of the participating laboratories, at the expense of the participant who carried out the transport. A safe transport was realized by all the participants.

The individual calibration laboratories generally adhered to the assigned time frame for their measurements. In some cases, delays occurred. These delays were then coordinated with the pilot laboratory and the following participants.

5 Applied calibration methods / Standard measuring equipment

5.1 Setting of tasks

The guideline DAkkS-DKD-R 5-4 “Calibration of temperature block calibrators” [1] and the accredited procedures of the participants formed the basis of the interlaboratory comparison / pilot study. The following examinations should be performed according to the setting of tasks.

1. Determination of the display deviation of the calibrator compared to a calibrated thermometer / thermocouple at the following measuring points:

   600 °C, 960°C, 1200 °C

   - Realization with increasing and decreasing temperature (hysteresis determination)

2. Determination of the temporal stability at the specified measuring points.

3. Determination of the axial temperature stability along the boring hole of the measuring zone, at least at the following distances from the bottom of the hole: 0 mm, 20 mm, 40 mm.
   The length of the measuring sensor used (SPRT or PRT) or the type of thermocouple should be specified respectively. Information on the homogeneity should be given for the thermocouple.
   A specially manufactured profile thermocouple was used at PTB and in the pilot laboratory (ZMK), thus being able to provide more accurate statements about the axial homogeneity. The profile thermocouple is described in more detail in paragraph 5.2.

4. *Optional, if metrologically feasible (not mandatory)*
   Determination of the differences in temperature between the holes by means of a second thermometer / thermocouple which – in addition to the calibrated thermometer / thermocouple – twice changes the measuring point (hole). If possible, opposite holes should be used.
   - was not performed by any of the participants –

5. Determination of the temperature in the measuring zone at different loading *(optional)*
   - was not performed by any of the participants-

Further specification of the tasks:

Preparation of the calibration

The block calibrators are to be set up in such a way that a minimum distance of 30 cm is guaranteed on all sides. Above the calibrator, there should be at least one meter of free space. The insertion tube must be carefully placed by means of the special tool.

Precautions

Generally, the safety instructions specified in the manufacturer's operating documents are to be observed. The block calibrator must be free-standing, with the specified minimum distance required. The use of liquids, thermal conductance paste, etc. in the insertion tubes is not allowed.

Ambient conditions

The calibration should be performed at an ambient temperature of 23 °C (± 2 K). The ambient temperature during the measurement shall be documented.

Presentation of the measurement results

The measurement results should be presented in a calibration certificate according to DAkkS-DKD-5 [3].

To simplify the subsequent evaluation, the participants were also provided with an Excel spreadsheet in which the measurement results of the individual tests were entered.

As to the thermoelectric homogeneity of the thermocouple used, there should be a precise indication of how this homogeneity had been determined.

The participants transmitted their measurement results to PTB.

The pilot laboratory (ZMK) received the results of the other participants for evaluation only ***after*** *having transmitted its own measurement results of the initial measurement and the re-measurement to PTB*.

5.2 Standard devices used

The participants used various standard platinum resistance thermometers (SPRTs), platinum resistance thermometers and thermocouples for the individual examinations.

To carry out the interlaboratory comparison, a special profile thermocouple was manufactured by *Electrotherm Gesellschaft für Sensorik und thermische Meßtechnik mbH* according to specifications provided by PTB.

By using the profile thermocouple, the statements on the axial homogeneity / heat dissipation could be qualified.

The measurements at PTB and in the pilot laboratory (ZMK) were additionally performed with a profile thermocouple.

Fig. 2 shows the structure of the profile thermocouple.

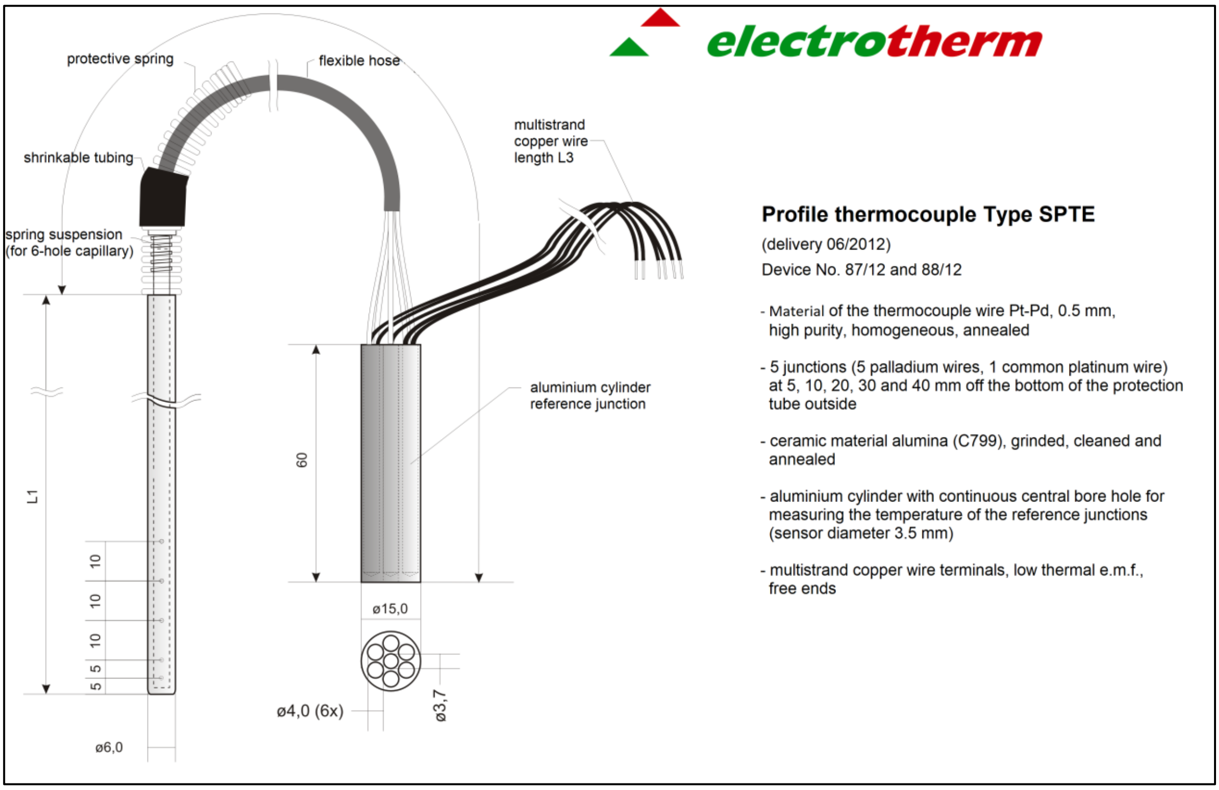


Fig. 2: Specially manufactured profile thermocouple

The use of different thermometers and thermocouples allowed a complex evaluation of the achieved measurement results.

Due to the use of different standards, it was also possible to assign achievable measurement uncertainties to the measurement results.

Table 3 shows an overview of the standards used.

Table 3: Overview of the standards used by the participants

Type of Standard	Type / Manufacturer	Application area / Measuring range	Diameter / Sensor length
Standard platinum resistance thermometer (SPRT)	Pt 25 / Rosemount	-200 °C to 661 °C	5.8 mm / 38 mm
	Pt 25 / Rosemount	-200 °C to 661 °C	5.8 mm / 38 mm
	Pt 25 / Rosemount	-200 °C to 661 °C	5.5 mm / 40 mm
	Pt 25 / 5698-25 / Fluke	-200 °C to 670 °C	7 mm / 40 mm
	Pt 25 / 5681 / Fluke	-200 °C to 670 °C	7 mm / 38 mm
High temperature standard platinum resistance thermometer (SPRT)	5624 / Fluke	0 °C to 1000 °C	6.4 mm / 45 mm
Platinum resistance thermometer (PRT)	Pt 100 / 5609 / Fluke	-200 °C to 670 °C	3 mm / 30 mm
Noble metal thermocouple	Typ Pt/Pd / electrotherm	0 °C to 1500 °C	7 mm / -
	Typ Pt/Pd / electrotherm	0 °C to 1500 °C	7 mm / -
	Typ Pt/Pd / electrotherm	0 °C to 1500 °C	6 mm / -
	Typ S / electrotherm	0 °C to 1450 °C	6.1 mm / -
	Typ S / Heraeus	0 °C to 1500 °C	7 mm / -
	Typ S / Heraeus	0 °C to 1500 °C	7 mm / -
	Typ S / CTP9000	0 °C to 1300 °C	7.1 mm / -
	Typ S/ 5650 / Fluke	0 °C to 1450 °C	6.4 mm / -
Profile thermocouple	Pt/Pd / electrotherm	0 °C to 1500 °C	7 mm / -
	Pt/Pd / electrotherm	0 °C to 1500 °C	7 mm / -

6 Results of the interlaboratory comparison / pilot study

The results were documented by the participants in calibration certificates. Due to a non-disclosure agreement, the numbers of the calibration certificate are not listed in the report. The following tables summarize the measurement results of the interlaboratory comparison. The stated measurement results are average values from multiple measurements. The participating laboratories were encrypted by laboratory codes.

It is the expanded measurement uncertainty which is indicated. The expanded uncertainty results from the standard measurement uncertainty by multiplication with the expansion factor *k = 2*. It was determined according to DAkkS-DKD-3 [4]. With a probability of approximately 95 %, the value of the measured variable lies within the assigned interval of values.

6.1 Measurement results of the participant with the laboratory code 01

Table 4: Determination of the display deviation – Laboratory 01

Measuring temperature in °C	Type of standard	Display / Measured value Standard * in °C	Display / Measured value Calibration item * in °C	Deviation in K	Expanded measurement uncertainty (k = 2) in K
Insert 1 // Hole diameter 7.5 mm					
600	SPRT	598.73	600.0	1.3	*Additional examination*
600	TE	598.62	600.0	1.4	2.5
960	TE	557.62	960.0	2.4	4.0
1200	TE	1199.07	1200.0	0.9	5.0

** Mean value from upwards and downwards measurements*

Table 5: Further test results according to DAkkS-DKD-R 5-4 – Laboratory 01

Measuring temperature in °C	Type of standard	Stability in K	Hysteresis in K	Axial homogeneity in K
Insert 1 // Hole diameter 7.5 mm				
600	SPRT	0.02	0.46	6.9
600	TE	0.06	0.36	-
600	STE	-	-	14.6
960	TE	0.03	1.6	-
960	STE	-	-	17.2
1200	TE	0.05	0.03	7.4
1200	STE	-	-	17.5

6.2 Measurement results of the participant with the laboratory code 02

Table 6: Determination of the display deviation – Laboratory 02

Measuring temperature in °C	Type of standard	Display / Measured value Standard * in °C	Display / Measured value Calibration item * in °C	Deviation in K	Expanded measurement uncertainty (k = 2) in K
Insert 1 // Hole diameter 7.5 mm					
600	SPRT	599.55	600.0	0.5	*Additional examination / not used for calibration certificate*
600	TE	598.01	600.0	2.0	2.4
960	TE	957.4	960.0	2.6	7.0
1200	TE	1197.71	1200.0	2.3	9.2

** Mean value from upwards and downwards measurements*

Table 7: Further test results according to DAkkS-DKD-R 5-4 – Laboratory 02

Measuring temperature in °C	Type of standard	Stability in K	Hysteresis in K	Axial homogeneity in K
Insert 1 // Hole diameter 7.5 mm				
600	SPRT	0.01	0.02	2.8
600	TE	0.02	0.05	4.1
600	STE	-	-	1.5
960	TE	0.01	0.04	6.7
960	STE	-	-	5.8
1200	TE	0.05	0.08	8.0
1200	STE	-	-	7.6

6.3 Measurement results of the participant with the laboratory code 03

Table 8: Determination of the display deviation – Laboratory 03

Measuring temperature in °C	Type of standard	Display / Measured value Standard in °C	Display / Measured value Calibration item * in °C	Deviation in K	Expanded measurement uncertainty (*k* = 2) in K
Insert 1 // Hole diameter 7.5 mm					
600	SPRT	598.72	600.0	1.3	4.6
600	TE	598.39	600.0	1.6	3.8
960	TE	957.79	960.0	2.2	8.3
1200	TE	1198.69	1200.0	1.3	10.2
Insert 2 // Hole diameter 6.5 mm					
600	SPRT	598.50	600.0	1.5	4.5
600	TE	598.87	600.0	1.1	2.6
960	TE	958.08	960.0	1.9	6.4
1200	TE	1198.86	1200.0	1.1	8.3

** Mean value from upwards and downwards measurements*

Table 9: Further test results according to DAkkS-DKD-R 5-4 – Laboratory 03

Measuring temperature in °C	Type of standard	Stability in K	Hysteresis in K	Axial homogeneity in K
Insert 1 // Hole diameter 7.5 mm				
600	SPRT	0.02	0.18	6.3
600	TE	0.19	0.12	9.5
960	TE	0.11	0.26	12.7
1200	TE	0.07	0.07	15.9
Insert 2 // Hole diameter 6.5 mm				
600	SPRT	0.13	0.12	6.4
600	TE	0.04	0.05	6.3
960	TE	0.06	0.24	12.2
1200	TE	0.04	0.00	15.1

6.4 Measurement results of the participant with the laboratory code 04

Table 10: Determination of the display deviation – Laboratory 04

Measuring temperature in °C	Type of standard	Display / Measured value Standard * in °C	Display / Measured value Calibration item * in °C	Deviation in K	Expanded measurement uncertainty (k = 2) in K
Insert 1 // Hole diameter 7.5 mm					
600	SPRT	599.02	600.0	1.0	*Additional examination / not used for calibration certificate*
600	SPRT	598.65	600.0	1.4	*Additional examination / not used for calibration certificate*
600	TE	599.64	600.0	0.4	3.6
960	SPRT	958.64	960.0	1.4	*Additional examination / not used for calibration certificate*
960	TE	959.47	960.0	0.6	6.4
1200	TE	1200.11	1200.0	-0.1	7.9

** Mean value from upwards and downwards measurements*

Table 11: Further test results according to DAkkS-DKD-R 5-4 – Laboratory 04

Measuring temperature in °C	Type of standard	Stability in K	Hysteresis in K	Axial homogeneity in K
Insert 1 // Hole diameter 7.5 mm				
600	SPRT	0.10	0.00	1.2
600	SPRT	0.05	0.05	0.9
600	TE	.,25	0.15	6.0
960	SPRT	0.26	0.00	2.5
960	TE	0.02	0.19	11.1
1200	TE	0.09	0.00	13.5

6.5 Measurement results of the participant with the laboratory code 05

Table 12: Determination of the display deviation – Laboratory 05

Measuring temperature in °C	Type of standard	Display / Measured value Standard * in °C	Display / Measured value Calibration item * in °C	Deviation in K	Expanded measurement uncertainty (k = 2) in K
Insert 1 // Hole diameter 7.5 mm					
600	SPRT	599.58	600.0	0.4	5.6
600	TE	598.03	600.0	2.0	*Additional examination / not used for calibration certificate*
960	TE	957.41	960.0	2.6	17.9
1200	TE	1198.13	1200.0	1.9	14.4

** Mean value from upwards and downwards measurements*

Table 13: Further test results according to DAkkS-DKD-R 5-4 – Laboratory 05

Measuring temperature in °C	Type of standard	Stability in K	Hysteresis in K	Axial homogeneity in K
Insert 1 // Hole diameter 7.5 mm				
600	SPRT	0.03	0.39	4.7
600	TE	0.09	0.15	4.7
960	TE	0.04	0.01	15.3
1200	TE	0.05	0.01	12.2

6.6 Measurement results of the participant with the laboratory code 06

Table 14: Determination of the display deviation – Laboratory 06

Measuring temperature in °C	Type of standard	Display / Measured value Standard * in °C	Display / Measured value Calibration item * in °C	Deviation in K	Expanded measurement uncertainty (k = 2) in K
Insert 2 // Hole diameter 6.5 mm					
600	TE	598.57	600.0	1.4	3.9
960	TE	957.37	960.0	2.6	7.6
1200	TE	1199.26	1200.0	0.7	9.1

** Mean value from upwards and downwards measurements*

Table 15: Further test results according to DAkkS-DKD-R 5-4 – Laboratory 06

Measuring temperature in °C	Type of standard	Stability in K	Hysteresis in K	Axial homogeneity in K
Insert 2 // Hole diameter 6.5 mm				
600	TE	0.04	0.17	6.2
960	TE	0.04	0.33	12.9
1200	TE	0.06	0.17	15.1

6.7 Measurement results of the participant with the laboratory code 07

Table 16: Determination of the display deviation – Laboratory 07

Measuring temperature in °C	Type of standard	Display / Measured value Standard * in °C	Display / Measured value Calibration item * in °C	Deviation in K	Expanded measurement uncertainty (k = 2) in K
Insert 1 // Hole diameter 7.5 mm					
600	TE	597.64	600.0	2.4	4.9
960	TE	957.10	960.0	2.9	8.0
1200	TE	1197.65	1200.0	2.3	9.8

** Mean value from upwards and downwards measurements*

Table 17: Further test results according to DAkkS-DKD-R 5-4 – Laboratory 07

Measuring temperature in °C	Type of standard	Stability in K	Hysteresis in K	Axial homogeneity in K
Insert 1 // Hole diameter 7.5 mm				
600	TE	0.06	0.08	4.1
960	TE	0.07	0.18	6.8
1200	TE	0.12	0.15	8.4

6.8 Measurement results of the participant with the laboratory code 08

Table 18: Determination of the display deviation – Laboratory 08

Measuring temperature in °C	Type of standard	Display / Measured value Standard * in °C	Display / Measured value Calibration item * in °C	Deviation in K	Expanded measurement uncertainty (k = 2) in K
Insert 1 // Hole diameter 7.5 mm					
600	SPRT	598.50	600.0	1.5	*Additional examination*
600	TE	597.83	600.0	2.2	2.5
960	TE	956.51	960.0	3.5	4.0
1200	TE	1196.82	1200.0	3.2	5.0
Insert 2 // Hole diameter 6.5 mm					
600	PRT	598.02	600.0	2.0	*Additional examination*
600	SPRT	598.35	600.0	1.7	*Additional examination*
600	TE	597.83	600.0	2.2	2.5
960	TE	956.90	960.0	3.1	4.0
1200	TE	1197.5	1200.0	2.5	5.0

** Mean value from upwards and downwards measurements*

Table 19: Further test results according to DAkkS-DKD-R 5-4 – Laboratory 08

Measuring temperature in °C	Type of standard	Stability in K	Hysteresis in K	Axial homogeneity in K
Insert 1 // Hole diameter 7.5 mm				
600	PRT	-	-	4.3
600	SPRT	0.02	0.00	6.1
600	TE	0.03	0.05	-
600	STE	-	-	2.3
960	TE	-	0.09	15.8
960	STE	-	-	6.7
1200	TE	0.02	0.01	13.4
1200	STE	-	-	10.0
Insert 2 // Hole diameter 6.5 mm				
600	PRT	0.01	0.01	2.8
600	SPRT	0.06	0.02	5.9
600	TE	0.04	0,08	7.2
600	STE	-	-	1.9
960	TE	0.08	0.04	13.6
960	STE	-	-	5.4
1200	TE	0.02	0.01	18.0
1200	STE	-	-	8.3

7 Determination of the axial homogeneity as a significant uncertainty contribution

Besides the display deviation, further parameters are determined during the calibration of block calibrators. These parameters serve to characterize the calibration item and are integrated into the consideration of the measurement uncertainty.

The axial homogeneity of the calibration item and the heat dissipation via the standard being used represent essential contributions. It is difficult to separate both contributions, so in the following they are simply referred to as axial homogeneity. The axial homogeneity is determined according to the guideline DAkkS-DKD-R 5-4 [1] along the hole of the insertion tube of the block calibrator.

As part of the pilot study, it was specified that the axial homogeneity had to be determined within a measuring zone of 40 mm length at the bottom of the holes.

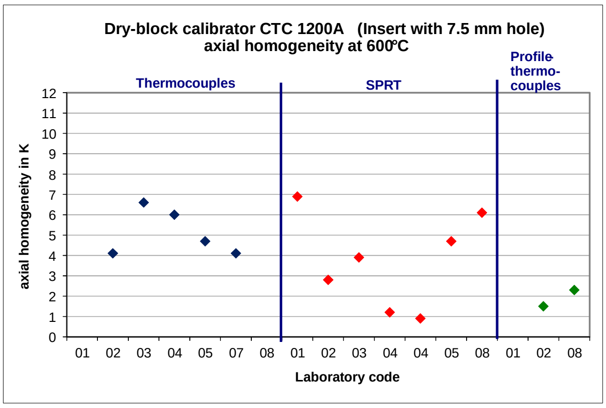
The participants used different types of standards to reflect as broad an image as possible of the everyday practice in calibration laboratories.

Table 20 shows the results of the participants in determining the axial homogeneity of the calibration item using different standards.

Table 20: Results of the participants for the determination of the axial homogeneity of the calibration item

Participant / Laboratory code	Type of standard / Insert used	Determined axial homogeneity at 600 °C in K	Determined axial homogeneity at 960 °C in K	Determined axial homogeneity at 1200 °C in K
01	SPRT / Insert 1	6.9	-	-
01	TC / Insert 1	-	-	-
01	Profile TC / Insert 1	-	-	-
02	SPRT / Insert 1	2.8	-	-
02	TC / Insert 1	4.1	6.7	8.0
02	Profile TC / Insert 1	1.5	5.8	7.6
03	SPRT / Insert 1	3.9	-	-
03	TC / Insert 1	6.6	14.4	17.7
03	SPRT / Insert 2	3.9	-	-
03	TC / Insert 2	4.3	11.0	14.2
04	SPRT / Insert 1	1.2	-	-
04	SPRT / Insert 1	0.9	2.6	-
04	TC / Insert 1	6.0	11.1	13.5
05	SPRT / Insert 1	4.7	-	-
05	TC / Insert 1	4.7	15.3	12.2
06	TC / Insert 2	6.2	12.9	15.1
07	TC / Insert 1	4.1	6.8	8.4
08	SPRT / Insert 1	6.2	-	-
08	TC / Insert 1	-	15.8	13.4
08	Profile TC / Insert 1	2.3	6.7	10.0
08	PRT / Insert 2	2.8	-	-
08	SPRT / Insert 2	5.9	-	-
08	TC / Insert 2	7.2	13.6	18.0
08	Profile TC / Insert 2	1.9	5.4	8.3

The following figures show the graphical representation of the test results regarding the axial homogeneity of the calibration item using the inserts 1 and 2 with different drill hole diameters.



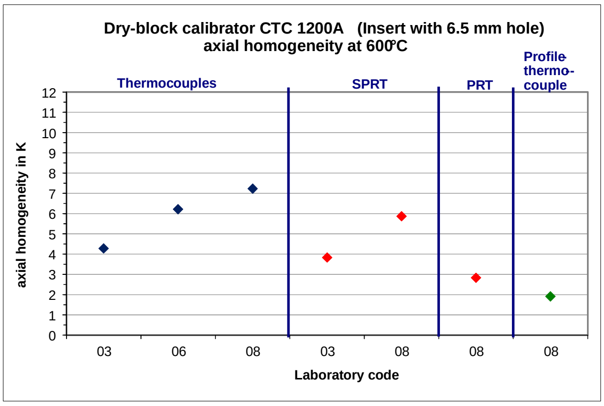
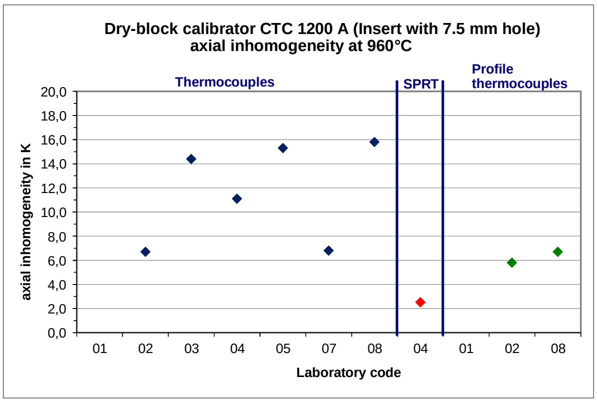


Fig. 3a / 3b: Graphical representation of the axial homogeneity determined by the participants at 600 °C in Insert 1 and Insert 2



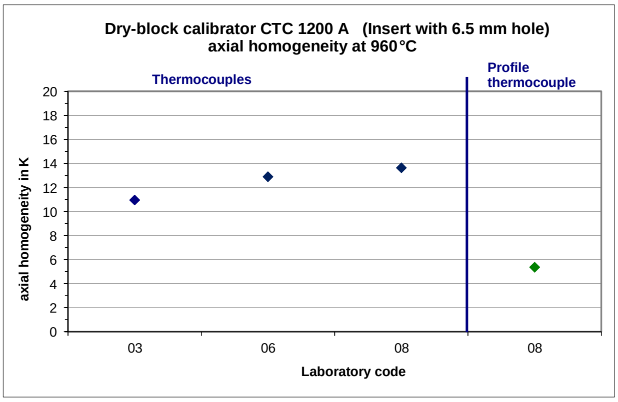
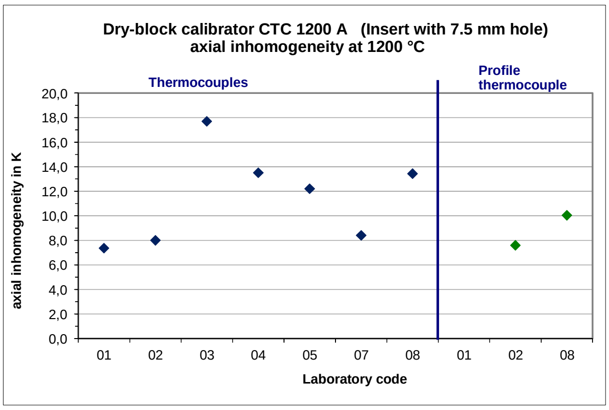


Fig. 4a / 4b: Graphical representation of the axial homogeneity determined by the participants at 960 °C in Insert 1 and Insert 2



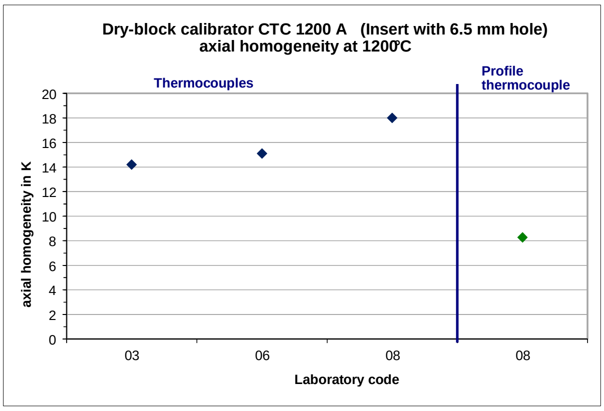


Fig. 5a / 5b: Graphical representation of the axial homogeneity determined by the participants at 1200 °C in Insert 1 and Insert 2

As a result of the investigations of the axial homogeneity, it became clear that the estimation of the so far accredited measurement uncertainties of the calibration laboratories had been very optimistic.

Based on the results of the interlaboratory comparison / pilot study, the following lower limits for the contribution to the axial homogeneity of high-temperature block calibrators for temperatures above 600 °C were determined (Table 21). The contribution to the axial homogeneity is to be considered in the uncertainty budget.

Table 21: Minimum values for the contribution of the axial homogeneity to be considered in the uncertainty budget
(for high-temperature block calibrators for temperatures above 600 °C)

Measuring temperature in °C	Minimum value for the axial homogeneity of block calibrators t_{max} - t_{min} in K
600	2.0
960	6.0
1200	8.0

According to the guideline DAkkS-DKD-R 5-4 [1], the contribution of the axial homogeneity is to be considered in the uncertainty budget as follows:

$$u_i(t) = \frac{t_{\max} - t_{\min}}{2 \cdot \sqrt{3}} \quad (1)$$

As a precondition for this procedure, at least three individual measurements must have been performed, i.e. measurements with at least three immersion depths over a length of at least 4 cm.

8 Evaluation of the interlaboratory comparison / pilot study

For the evaluation of the interlaboratory comparison / pilot study, the display deviations determined by the participants and the associated measurement uncertainties were considered in comparison to the reference value and its measurement uncertainty.

The reference value (best estimate) X_{Ref} was calculated from the weighted average of the results of all participants $x_{i,Lab}$.

$$X_{\mathrm{Ref}} = \frac{\sum_{i=1}^{n} \frac{x_{i,Lab}}{u_i^2}}{\sum_{i=1}^{n} \frac{1}{u_i^2}} \tag{2}$$

The interlaboratory comparison was carried out with two insertion tubes (Insert 1 and 2) with different bore diameters (7.5 mm and 6.5 mm).
The mean values of the participant results with Insert 1 and Insert 2 showed a maximum difference of 0.2 K. Therefore, the weighted average of all results, irrespective of the insert used, was taken as the reference value.

The standard measurement uncertainty of the reference value $u_{X_{\mathrm{Ref}}}$ was calculated according to equation (3) from the standard uncertainties of the participants u_i [5].

$$\frac{1}{u_{X_{\mathrm{Ref}}}^2} = \frac{1}{u_1^2} + \ldots + \frac{1}{u_n^2} \tag{3}$$

The measurement results of the participants, the reference values derived thereof at the individual measuring temperatures and the associated expanded measurement uncertainties are set out in Table 22.

Table 22: Measurement results of the participants and reference values calculated on this basis with associated measurement uncertainties

Participant / Laboratory code	Measuring temperature: 600 °C		Measuring temperature: 960 °C		Measuring temperature: 1200 °C	
	Detected deviation at 600 °C in K	Expanded measurement uncertainty (*k* = 2) in K	Deviation at 960 °C in K	Expanded measurement uncertainty (*k* = 2) in K	Deviation at 1200 °C in K	Expanded measurement uncertainty (*k* = 2) in K
01	1.4	2.5	2.4	4.0	0.9	5.0
02	2.0	2.4	2.6	7.0	2.3	9.2
03	1.1	2.6	1.9	6.4	1.1	8.3
03	1.6	3.8	2.2	8.3	1.3	10.2
04	0.4	3.6	0.6	6.4	-0.1	7.9
05	0.4	5.6	2.6	17.9	1.9	14.4
06	1.4	3.9	2.6	7.6	0.7	9.1
07	2.4	4.9	2.9	8.0	2.3	9.8
08	2.2	2.5	3.5	4.0	3.2	5.0
08	2.2	2.5	3.1	4.0	2.6	5.0
Reference values with expanded measurement uncertainties						
	600 °C		**960 °C**		**1200 °C**	
	Mean value of the deviation in K	Expanded measurement uncertainty *(k=2)* in K	Mean value of the deviation in K	Expanded measurement uncertainty *(k=2)* in K	Mean value of the deviation in K	Expanded measurement uncertainty *(k=2)* in K
	1.6	**1.0**	**2.6**	**1.9**	**1.8**	**2.3**

The E_n value, which represents the relevant quality criterion, was calculated for the evaluation of the interlaboratory comparison / pilot study.

The E_n value is the normalized difference to the expanded measurement uncertainty between a measured value and its reference value normalized and is calculated according to the following formula (4):

$$E_n = \frac{x_{lab} - x_{ref}}{\sqrt{(U_{lab}^2 + U_{ref}^2)}} \tag{4}$$

x_{lab}...... Measured valued of the participating calibration laboratory

x_{ref}...... Reference value

U_{lab}..... Expanded measurement uncertainty (k = 2) of the participating calibration laboratory

U_{ref}..... Expanded measurement uncertainty (k = 2) of the reference value

Tables 23 to 25 summarize the results of the participants in comparison to the reference values, including the E_n value.

Figures 6 to 8 provide a graphical representation of the results. The continuous red line represents the reference value. The broken lines represent the expanded measurement uncertainty of the reference value.

Table 23: Summary of the results for determining the deviation of the calibration item at a measuring temperature of 600 °C

Participant / Laboratory code	Type of standard / insert used	Result of the participant		Reference value		| E_n |
		Detected deviation at 600 °C in K	Expanded measurement uncertainty (k = 2) in K	Deviation at 600 °C in K	Expanded measurement uncertainty (k = 2) in K	
01	TC / Insert 1	1.4	2.5	1.6	1.0	0.07
02	TC / Insert 1	2.0	2.4	1.6	1.0	0.15
03	TC / Insert 1	1.6	3.8	1.6	1.0	0.18
03	TC / Insert 2	1.1	2.6	1.6	1.0	0.00
04	TC / Insert 1	0.4	3.6	1.6	1.0	0.32
05	SPRT / Insert 1	0.4	5.6	1.6	1.0	0.21
06	TC / Insert 2	1.4	3.9	1.6	1.0	0.05
07	TC / Insert 1	2.4	4.9	1.6	1.0	0.16
08	TC / Insert 1	2.2	2.5	1.6	1.0	0.22
08	TC / Insert 2	2.2	2.5	1.6	1.0	0.22

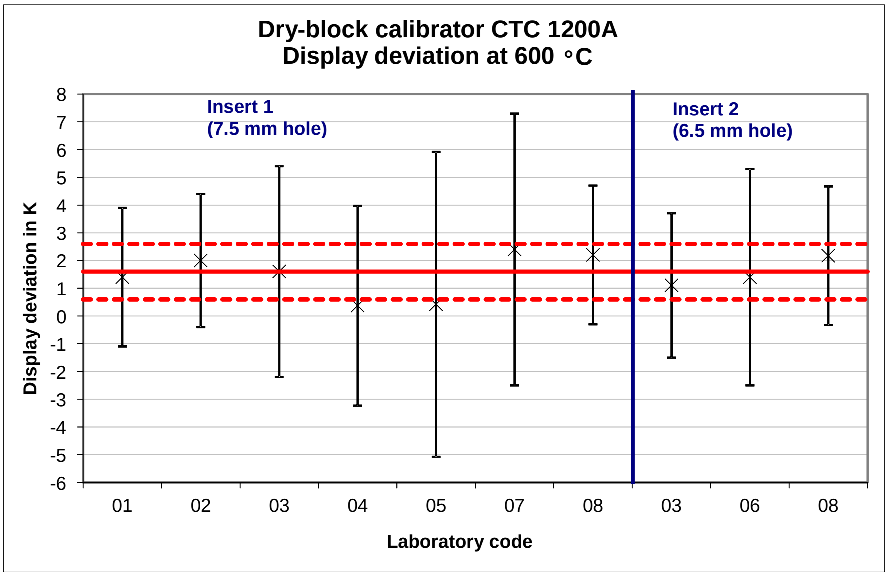


Fig. 6: Graphical presentation of the results for determining the deviation of the calibration item at a measuring temperature of 600 °C

Table 24: Summary of the results for determining the deviation of the calibration item at a measuring temperature of 960 °C

Participant / Laboratory code	Type of standard / insert used	Result of the participant		Reference value		| E_n |
		Detected deviation at 960 °C in K	Expanded measurement uncertainty (k = 2) in K	Deviation at 960 °C in K	Expanded measurement uncertainty (k = 2) in K	
01	TC / Insert 1	2.4	4.0	2.6	1.9	0.05
02	TC / Insert 1	2.6	7.0	2.6	1.9	0.00
03	TC / Insert 1	2.2	8.3	2.6	1.9	0.10
03	TC / Insert 2	1.9	6.4	2.6	1.9	0.05
04	TC / Insert 1	0.6	6.4	2.6	1.9	0.30
05	TC / Insert 1	2.6	17.9	2.6	1.9	0.00
06	TC / Insert 2	2.6	7.6	2.6	1.9	0.00
07	TC / Insert 1	2.9	8.0	2.6	1.9	0.04
08	TC / Insert 1	3.5	4.0	2.6	1.9	0.20
08	TC / Insert 2	3.1	4.0	2.6	1.9	0.11

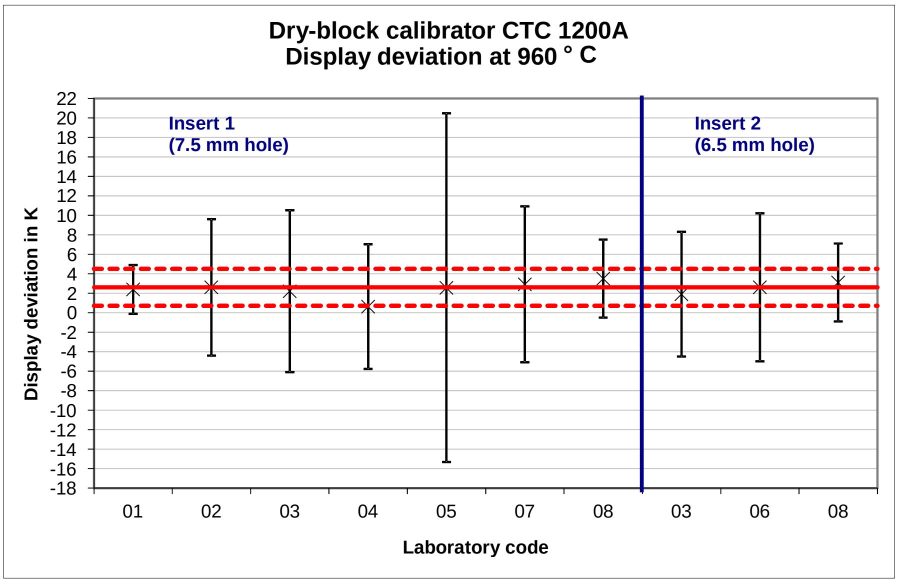


Fig. 7: Graphical presentation of the results for determining the deviation of the calibration item at a measuring temperature of 960 °C

Table 25: Summary of the results for determining the deviation of the calibration item at a measuring temperature of 1200 °C

Participant / Laboratory code	Type of standard / insert used	Result of the participant		Reference value		$\lvert E_n \rvert$
		Detected deviation at 1200 °C in K	Expanded measurement uncertainty (k = 2) in K	Deviation at 1200 °C in K	Expanded measurement uncertainty (k = 2) in K	
01	TC / Insert 1	0.9	5.0	1.8	2.3	0.16
02	TC / Insert 1	2.3	9.2	1.8	2.3	0.05
03	TC / Insert 1	1.3	10.2	1.8	2.3	0.08
03	TC / Insert 2	1,1	8.3	1.8	2,3	0.05
04	TC / Insert 1	-0.1	7.9	1.8	2.3	0.23
05	TC / Insert 1	1.9	14.4	1.8	2,3	0.00
06	TC / Insert 2	0.7	9.1	1.8	2.3	0.12
07	TC / Insert 1	2.3	9.8	1.8	2.3	0.05
08	TC / Insert 1	3.2	5.0	1.8	2,3	0.25
08	TC / Insert 2	2.6	5.0	1.8	2.3	0.14

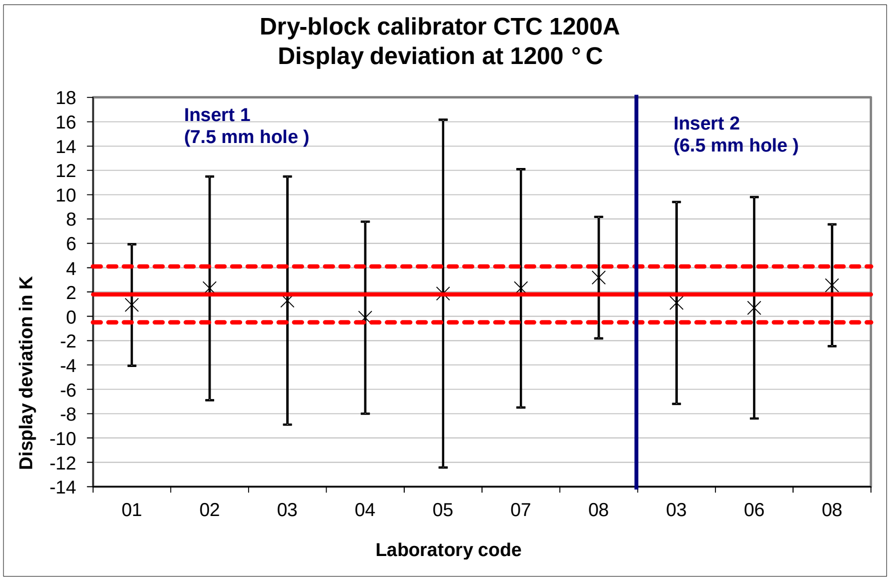


Fig. 8: Graphical presentation of the results for determining the deviation of the calibration item at a measuring temperature of 1200 °C

The determined absolute values of the E_n values of the comparison measurement are smaller than 1, i.e. the measurement results of the participants match within the scope of the indicated expanded measurement uncertainties.

This is based on the measurement uncertainties calculated by the participants which among other things include the experimentally determined axial homogeneity as an essential uncertainty contribution.

The results are to be kept ***confidential*** and shall only be used in the context of the interlaboratory comparison / pilot study.

The pilot laboratory would like to thank all participants for their participation and cooperation.

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