

Physikalisch- Technische Bundesanstalt



**Expert Report
DKD-E 5-1**

**Additions to guidelines
DKD-R 5-1:2018, DKD-R 5-3:2018
and DKD-R-5-4:2018
General recommendations and
recommendations on comparisons**

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Deutscher Kalibrierdienst (DKD) – German Calibration Service

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

This body is known as *Deutscher Kalibrierdienst* (DKD for short) and is under the direction of PTB. The guidelines and guides developed by DKD represent the state of the art in the respective areas of technical 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 supervised by DAkkS as legal successor to the DKD. They carry out calibrations of measuring instruments and measuring standards for the measurands 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

DKD expert reports aim to provide background information and references in connection with other DKD documents as, for example, the DKD guidelines. In some cases, they may even go far beyond these documents. They do not replace the original DKD documents but do provide a lot of supplementary information worth knowing. The expert reports do not necessarily reflect the views of the DKD's Management Board or Technical Committees in all details.

DKD expert reports are intended to present significant aspects from the field of calibration. Through publication by the DKD they are made available to the large community of calibration laboratories, both nationally and internationally.

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1 Introduction

The primary task of this expert report is to provide guidance to calibration laboratories on various topics in the field of thermodynamic measurands. Although these topics have already been discussed within DKD's Technical Committee *Temperature and Humidity* as well as in the DKD Guidelines, binding regulations have not yet been established. Hence, this report aims to ensure adaptation to the international state of the art as well as a consistent approach to strengthen the competence of the accredited calibration laboratories.

The following chapters provide supplementary information and recommendations regarding the calibration of resistance thermometers according to DKD-R 5-1 [1], the calibration of thermocouples according to DKD-R 5-3 [2] as well as the recalibration intervals of reference standards and the axial temperature distribution in block calibrators (DKD-R 5-4 [6]).

Another essential point is the subject of comparison measurements. The report offers recommendation concerning the documentation of proficiency testing / comparison measurements presenting a result indicator by which the significance of comparison measurements can be uniformly assessed.

2 Additions to DKD-R 5-1:2018 Calibration of resistance thermometers

For temperature measurements in industry, usually industrial resistance thermometers (IPRTs) – such as Pt-100 sensors – are used. There are various types of sensors and accuracy classes to choose from, depending on the measurement task. With these thermometers, it is possible to reach uncertainties within ± 10 mK in the temperature range from -40 °C to 100 °C, and ± 50 mK in the temperature range from -80 °C to 662 °C (exclusive determination of characteristic curve) [3]. In addition to the influence quantities caused by the calibration procedure and calibration equipment, the two properties self-heating and hysteresis constitute essential uncertainty components. Their handling and determination are described here in more detail as an addition to the guideline DKD-R 5-1 [1].

2.1 Self-heating

Since resistance thermometers are passive components, an electric current must be sent through the sensor to determine the electrical resistance. The measuring current causes the sensor to be heated (self-heating). If no appropriate measures are taken to correct this influence, this effect will lead to greater uncertainties in the measurement result. The self-heating effect does not only depend on the magnitude of the measuring current, but also on the measuring conditions (thermal coupling) and the design of the sensor.

When calibrating resistance thermometers, the self-heating behaviour must be investigated. Otherwise, the influence is to be estimated.

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To determine the self-heating, the sensor resistance is usually measured using two different measuring currents. The resistance value for the theoretical measuring current intensity I of 0 mA (or the electrical power P to 0 mW) is then determined by extrapolation. This “0 mA value” allows the thermometer to be used under any conditions; however, the influence of the respective conditions has to be determined or estimated by the user. [3, 7, 8]

If, for instance, it is not possible to change the intensity of the measuring current, another possibility would be to adjust the thermal coupling of the thermometer to its environment. An effective solution consists in placing a small tube (made of glass, for example) with a large diameter (compared to the sensor diameter) in a temperature-homogeneous environment sufficiently stable over a period of time (e.g. ice point). Subsequently, a measurement is carried out in the lower part of the tube using the distinctly smaller sensor. The sensor is surrounded completely by air and must not touch the wall of the tube. The tube is then filled with a contact medium of good thermal conductivity (aluminium oxide, for example) until the entire sensor is surrounded by it. After an adequate stabilisation time, another measurement is then carried out, taking into account the different thermal expansions to avoid damage. The difference between the two measurement results can now be used to estimate the influence of self-heating for this extreme case between very good thermal coupling (aluminium oxide) and very poor coupling (air). Below the point of transition from tempering medium to environment, the tube must always be sealed (for example, with cotton wool) so that an undisturbed, stationary and homogeneous temperature distribution can form inside. Attention must be paid to possible heat dissipation.

In the calibration certificate, the results of the self-heating test must be stated in such a way to provide the users with all the necessary information to make an appropriate correction or to estimate an uncertainty for their conditions.

Where self-heating due to the measuring current is not determined experimentally, this contribution is to be taken into account in the uncertainty budget with 30 mK (rectangular distribution) for all types of resistance thermometers; this also applies when used in measuring chains. Assuming an asymmetrical rectangular distribution with the half-width a being 30 mK, the standard uncertainty is

$$u = \sqrt{\frac{1}{3} \cdot a^2} \approx 17 \text{ mK.} \quad (1)$$

If a certain type of thermometers is calibrated at regular intervals, it is possible to use the determined maximum values as reference for their self-heating – always provided that an adequate number of thermometer calibrations have been carried out (at least 10 thermometers of the same type). The thus determined typical self-heating of a thermometer type has to be verified at regular intervals by means of measurements / intermediate tests. This procedure must be described according to standards. Furthermore, the calibration certificate must refer to the fact that the value has not been explicitly determined for the thermometer to be calibrated.

2.2 Hysteresis

It should generally be noted that resistance thermometers, especially industrial platinum resistance thermometers (IPRT), show a hysteresis effect, i.e. the relationship between temperature and resistance depends on the thermal “history” of the thermometer. Such an effect occurs, for example, when the platinum sensor is closely connected to a glass or ceramic substrate resulting in mechanical stresses due to different thermal expansion (e.g. strain gauge effect with thin-film sensors on Al₂O₃ substrate).

In the case of resistance thermometers, this may cause a considerable difference in the measured resistance value even when used at the same temperature, depending on whether the thermometer has previously been used at higher or lower temperatures.

The hysteresis effect depends on the design of the sensor element. Experience shows that the effect is significantly stronger with glass-encapsulated PRTs and thin-film sensors; it can only be neglected when using standard platinum resistance thermometers (SPRTs), which is due to their special design. Usually, the greatest difference between the maximum and minimum values is to be found in the middle of the temperature range in which the sensor is used (Figure 1).

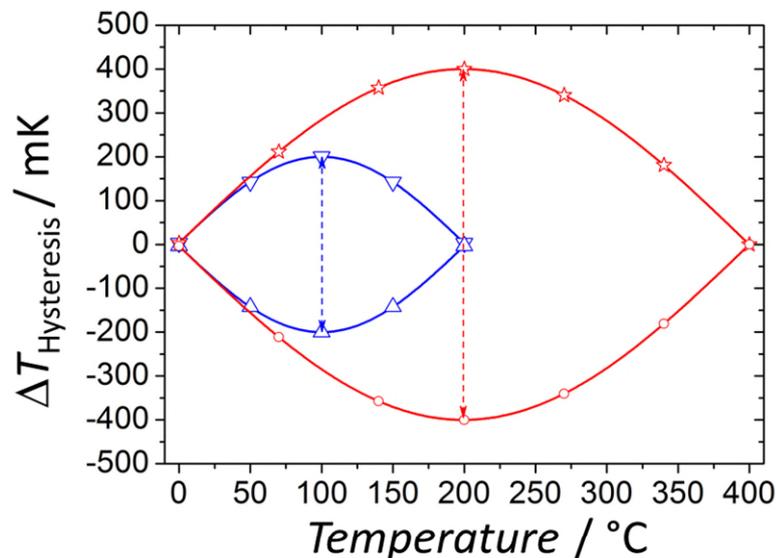


Figure 1: Theoretical examples for hysteresis-related characteristic curve deviations of an industrial resistance thermometer in different operating temperature ranges following [9-11]

The relevant literature shows that the error caused by hysteresis is between 0.002 % of the operating temperature range for the best ceramic encapsulated sensors and up to 0.2 % of the measuring range in °C for glass encapsulated sensors [9-11]. Therefore, a value of 0.2 % of the operating temperature range is to be applied in case of non-determination, as may be the case in practice.

It has been agreed by DKD’s Technical Committee *Temperature and Humidity* that this value does not necessarily have to be taken into account in the measurement

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uncertainty budget given that consistent handling does not exist at the international level.

It is therefore mandatory that the calibration certificates for resistance thermometers contain information regarding the influence of the hysteresis as well as the calibration conditions (calibration with ascending/descending temperatures). If the influence of the hysteresis is to be determined experimentally, the procedure must be in accordance with DIN EN 60751 [12] (Figure 1). In this case, calibration is carried out with ascending temperatures, i.e. starting at the lowest temperature. The most important calibration point for determining the hysteresis is located in the middle of the temperature range. After reaching the highest temperature, another measurement is carried out at the temperature lying in the middle of the calibration range.

Simplified measuring scheme without further calibration points of a direct reading thermometer at the measured temperatures t_n :

Lowest temperature (t_1) → mean temperature (t_2) →
 highest temperature (t_3) → directly to mean temperature (t_4)

$$\Delta t_{\text{Hysteresis}} = t_4 - t_2 \quad (2)$$

If the influence on the calibration result due to hysteresis is not determined experimentally, the effect is to be assumed to be 0.2 % of the total calibration range of the thermometer. The determined value is to be taken into account either in the measurement uncertainty budget or to be stated separately in the calibration certificate.

Example of a note in the calibration certificate in case the influence has not been determined experimentally:

“The calibration has been carried out in a temperature range from 0 °C to 400 °C using a programme with ascending temperature steps. The influence of the hysteresis has not been determined experimentally. In the above-mentioned temperature range, it can amount to up to 0.8 K if the measurements are not carried out continuously at ascending temperatures. The strongest influence is to be expected in the middle of the temperature range.”

The procedure described here shows its limits in case a change of the tempering device is necessary between the lowest and the highest temperature. To still be able to adequately determine the hysteresis, it is possible to determine a relative hysteresis which can then be used to draw conclusions about the entire temperature range.

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3 Additions to DKD-R 5-3:2018 Calibration of thermocouples: Inhomogeneity

The thermovoltage-temperature characteristic of thermocouples can change simply by using them under different thermal conditions. This also includes changing measurement uncertainties. Hence, for longer-term use of thermocouples, a procedure for periodic inspection and potential replacement of the thermocouples should be established. For base metal thermocouples used at high temperatures, replacement rather than recalibration is recommended [4].

When calibrating thermocouples [2, 4, 5], it is therefore important to consider the measurement uncertainty contribution due to thermoelectric inhomogeneity. The uncertainty contribution should be determined experimentally, ideally over the entire length of the thermocouple, but at least over a length of 10 cm. Given that the users must have all the necessary information – to be able to estimate, for example, a further uncertainty for their conditions – it is necessary to indicate in the calibration certificate the area of the thermocouple in which the inhomogeneity test has been carried out (e.g. 20 cm to 35 cm, measured from the tip of the thermocouple). Depending on the examined length of the thermocouple, the estimation of the measurement uncertainty contribution varies. If the examination has been carried out over (almost) the entire length of the thermocouple, a rectangular distribution is assumed; if, on the other hand, a much shorter section is examined, maxima and minima (peak-to-peak) should be used. In case the inhomogeneity contribution has not been determined experimentally, it has to be included in the measurement uncertainty budget using the contributions given in Table 1 (only for new thermocouples) from the EURAMET Guide No. 8 (Guidelines on the Calibration of Thermocouples, [4]) (relative standard uncertainty u with respect to temperature).

Type of thermocouples (new)	u
Type K and type N	0.10 %
Type R and type S	0.02 %
Type B	0.05 %
Au/Pt and Pt/Pd	0.01 %
Other types of thermocouples	0.25 %

Table 1: Uncertainty contributions for the inhomogeneity thermocouples [4]

The contribution of inhomogeneity of the calibration item can only be neglected if its calibration takes place in situ, i.e. with unchanged mounting. This applies to noble as well as base-metal thermocouples.

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4 Recommendations covering more than one guideline

The recommendations covering more than one guideline include the topics “Number and recalibration interval of reference standards (resistance thermometers, thermocouples)” and “Lower limits for the contribution of the axial temperature distribution of block calibrators”.

4.1 Number and recalibration interval of reference standards (resistance thermometers, thermocouples, etc.)

When using resistance thermometers or thermocouples as reference standards, there should generally be two standards (or more). The recommended maximum recalibration interval for these standards is 2 years. Depending on the measurement uncertainties to be achieved and the information about the long-term behaviour (drift, instability), the interval may be extended in certain cases to 3 years, with statement of reasons. To determine the start and end of the recalibration interval, the day of calibration is decisive. The day of calibration is decisive for the start and end of the recalibration interval. If a thermometer has been calibrated over a certain period of time, the last day of the specified interval counts.

The recommended maximum recalibration interval for fixed point cells is 5 years.

If intermediate checks reveal a change in the reference value of the reference standard by a significant amount (in relation to the uncertainty of the calibration according to the calibration certificate of the standard, e.g. $\geq 50\%$), the recalibration interval is to be adequately reduced. The specific amount is to be determined by the calibration laboratory based on its measurement uncertainty budget.

4.2 Lower limits for the contribution of the axial temperature distribution of block calibrators

In various laboratories, high-temperature block calibrators are on the one hand calibrated according to DKD-R 5-4 and on the other hand first characterised for the calibration of temperature sensors of various designs and then used as tempering devices.

The determination of the uncertainty contribution regarding the axial temperature distribution in block calibrators – especially for instruments designed for temperatures above 700 °C – proves to be rather complex due to different heat transport mechanisms, thermal couplings (block to furnace, thermometer to block), control characteristics and the feedback effect of the thermometer.

Therefore, the lower limits for the measurement uncertainty contribution of the axial temperature distribution in the block calibrator described below have been determined based on the pilot study “Calibration of temperature block calibrators at temperatures above 600 °C / Pilot study to determine the measurement uncertainty” [13]. Unless the lower limits have been investigated in detail (with thermometers of different types and

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designs) and documented, the contributions according to Table 2 must be taken into account as minimum contribution in the uncertainty budget for high-temperature block calibrators ($t_{\max} > 700 \text{ }^{\circ}\text{C}$).

Temperature range	Contribution / K	u / K	U ($k = 2$)
600 °C ... 800 °C	±2.0	1.2	2.5
> 800 °C ... 1000 °C	±3.4	2	4
> 1000 °C ... 1200 °C	±4.0	2.3	5
> 1200 °C ... 1300 °C	±5.0	2.9	6

Table 2: Indication of temperature-independent minimum uncertainty contributions with regard to the axial temperature inhomogeneity, taking into account a rectangular distribution and the resulting best measurement uncertainties

To take account of the rectangular distributed uncertainty contribution which depends on the temperature range, the related smallest expanded measurement uncertainties U (as part of the Calibration and Measurement Capabilities, CMCs) are indicated. These measurement uncertainties are based on the assumption that all other uncertainty components are negligible compared to the axial temperature distribution. However, using the above stated uncertainty contributions does not mean that the laboratories do not need to determine the axial temperature distribution for at least 3 immersion depths (exceeding 40 mm). If the determined contribution is greater than the value given in the table, then the determined contribution is to be used. The range of the 3 immersion depths constitutes the “homogeneous zone” of the block calibrator and must be stated in the calibration certificate.

5 Recommendation for the documentation of proficiency testing / comparison measurements

To demonstrate their performance, calibration laboratories must regularly compare their results with those of other calibration laboratories. For this purpose, offers are available from accredited proficiency testing providers whose competence has been demonstrated in accordance with DIN EN ISO/IEC 17043:2010 [14]. Participation in interlaboratory comparisons is accepted; however, this does not constitute a proficiency test in the strict sense (DIN EN ISO/IEC 17025:2018, point 7.7.2 [15]). Further information can be found in the DAkkS document “Use of proficiency testing in accreditation” (71 SD 0 010, [16]). Basically, this document describes the requirements for participation in proficiency tests within the scope of accreditation of conformity assessment bodies (CABs) and thus contributes to a harmonised application by assessors across all disciplines.

To harmonise the documentation of metrological comparisons other than proficiency tests and to increase their acknowledgement by the accreditation body, guidance and templates for the technical protocol / terms of reference and the results report are given below. These templates are based on the international recommendations of the Consultative Committee for Thermometry (CCT), document “CCT Guidelines for comparisons” [15] and are to be seen as a suggestion. In principle, it is advisable to agree on the procedure with the responsible process manager of the accreditation body before starting a metrological comparison and to fix everything in the technical

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protocol. Potential problems regarding the acknowledgement/acceptance of the comparisons in the accreditation procedure shall be avoided.

5.1 Assessment of proficiency testing / comparison measurements

Assessment of comparison measurements is usually calculated by means of the E_n number (result indicator) according to DIN EN ISO/IEC 17043:2010 (B.3.1.3.e) [14]; here we take a temperature measurement as an example:

$$E_n = \frac{t_{\text{lab}} - t_{\text{ref}}}{\sqrt{U_{\text{lab}}^2 + U_{\text{ref}}^2}} \quad (3)$$

with t_{lab} being the measurement result of a participating laboratory, t_{ref} the reference value, U_{lab} the expanded uncertainty of a participant and U_{ref} the expanded measurement uncertainty of the reference value. The reference value should be derived from multiple measurements. Assessment of the E_n value is as follows:

$$\begin{aligned} |E_n| \leq 1 &\rightarrow \text{successful (requirements fulfilled)} \\ |E_n| > 1 &\rightarrow \text{not successful (requirements not fulfilled)} \end{aligned}$$

Experience has shown that the choice of the transfer standard / calibration item does not always meet the stability requirements necessary to confirm the smallest measurement uncertainties. To take account of a potential drift of the transfer standard, the equation for determining the E_n value has been extended by the term Δt_{TS} . On the one hand, the drift can be determined by a backward measurement or, on the other hand, by means of specified tests (e.g. initial control measurement at the water triple point / ice point). Determination of the drift is not only essential for thermodynamic quantities, but also for all other physical and chemical measurands. The extension results in the new result indicator C_n , which takes into account the reliability of the measurement and the stability of the transfer standard itself:

$$C_n = \frac{\sqrt{\frac{1}{3}\Delta t_{\text{TS}}^2 + U_{\text{ref}}^2 + (t_{\text{lab}} - t_{\text{ref}})^2}}{U_{\text{lab}}} \quad (4)$$

with $C_n = |E_n|$ for $\Delta t_{\text{TS}} \ll U_{\text{lab}}$ and $U_{\text{ref}} \ll U_{\text{lab}}$. Care should be taken to ensure that – as shown above – the expanded measurement uncertainty of the reference value is significantly smaller than that of the participating calibration laboratory.

The evaluation of the C_n value is carried out in addition to the E_n value. It is carried out according to the following scheme:

$$\begin{aligned} C_n \leq 1 \text{ und } |E_n| \leq 1 &\rightarrow \text{successful} \\ C_n > 1 \text{ und } |E_n| > 1 &\rightarrow \text{not successful} \\ C_n > 1 \text{ und } |E_n| \leq 1 &\rightarrow \text{no adequate statement to confirm the CMCs} \end{aligned}$$

- Potential reasons for a C_n value greater than 1:

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- an E_n value near 1,
- a significant drift of the transfer standard and
- an inappropriately large measurement uncertainty of the reference value.

It is therefore strongly recommended to determine the C_n value if there are indications that might question the reliability of the comparison measurement. This allows for quantifiable, objective and reliable statements regarding the comparison measurement, thus helping to minimise the risk of unequal treatment between the laboratories. This also helps to support the acceptance of the comparison measurement as a proof of competence for the CMCs.

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5.2 Template: Technical protocol

Comparison of ...

Technical protocol
Main authors and affiliation
Date:
Version:

1. Introduction

- Initiator of the comparison measurement
- Objective, measurand and scope of the comparison measurement
- Reference documents for the creation of documents

2 Participants

- List of participating laboratories (contact persons, their postal and electronic addresses can be listed in a separate annex)
- Distribution of tasks (author of the technical protocol, pilot laboratory, trustee, evaluator, etc.)

3 Methodology of the comparison

- Topology of the comparison (bilateral, sequential, star, etc.)
- Start date and detailed schedule

4. Transfer standard / object of comparison

- Detailed description of the transfer standard (make, type, serial number, size, weight, packaging, ... and technical data necessary for operation)
- Instructions for handling the transfer standard, including packing instructions and dispatch to the next participant
- Tests to be carried out on the transfer standard after receipt and before measurement (e.g. measurement at the ice point / water triple point)
- Conditions regarding the use of the transfer standard during measurement
- Final tests before packing the transfer standard and sending it to the next laboratory
- Instructions on how to proceed in the event of a defect of the transfer standard

5. Organisational issues

- Procedure in case of unexpected delay of a participant
- Customs formalities and documents that must accompany the transfer standard on dispatch (ATA carnet or other documents)
- Financial aspects: Responsibility for the costs of the transfer standard, expenses for transportation, customs duties, costs in case of damage, insurance of the transfer standard

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6. Communication

- Participant to pilot laboratory: information about the receipt of the transfer standard
- Participant to pilot laboratory: notification about delayed measurements
- Participant to participant: notification of the next participant about the dispatch of the transfer standard
- Participant to pilot / trustee: reporting of measurement results
- Deadlines and consequences in case of non-compliance with deadlines

7. Measurement instructions and procedures

- Measurement instructions (indicate if there are special instructions)
- Measurement procedures (indicate if there are special procedures)

8. Reporting of results

- Instructions for reporting the results of tests to be performed after receipt of the transfer standard and prior to the actual measurement
- Instructions for reporting the measurement results (e.g. calibration certificates, Excel® spreadsheet)
- Instructions for stating the uncertainty of measurement (e.g. list of uncertainty components)
- Instructions for reporting additional information

9. Evaluation

- Method for determining the reference value, information regarding the evaluation

10. History of changes made in the document

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5.3 Template: Report

Comparison of ...

Report

Main authors and affiliation

Date:

Version:

1. Introduction

- Objective, measurand and scope of the comparison
- Brief summary of the comparison (the comparison started on..., the protocol was approved on..., the measurements were performed between... and... and...)

2 Participants

- List of participating laboratories (contact persons, their postal and electronic addresses can be listed in a separate annex)
- Distribution of tasks (author of the technical protocol, pilot laboratory, trustee, evaluator, etc.)

3 Methodology of the comparison

- Topology of the comparison (bilateral, sequential, star, etc.)

4. Transfer standard / object of comparison

- Detailed description of the transfer standard (make, type, serial number, size, weight, packaging, ... and technical data necessary for operation)

5. Equipment and measurement conditions in the participating laboratories

- Specific measurement instructions or procedures (if available)
- Detailed description of the equipment and measurement conditions in the participating laboratories

6. Measurement results

- Measurement results of each participating laboratory, including the uncertainty of each participating laboratory (the full measurement uncertainty budgets must be reported, but can be placed in a separate annex)

7. Evaluation of results

- Determination of the bilateral equivalence (E_n value) between the participating laboratories (for all comparisons) and the C_n value, if necessary

8. Conclusions

- Final remarks (have the goals been achieved?)
- Recommendations for future comparisons

	Additions to guidelines DKD-R 5-1:2018, DKD-R 5-3:2018 and DKD-R 5-4:2018 General recommendations and recommendations on comparisons https://doi.org/10.7795/550.20220610	DKD-E 5-1	
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9. Annexes

- Approved protocol

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