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Calibrating slender thermocouples oneself

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ABSTRACT

A simple method for calibrating slender temperature sensors like mineral insulated metal sheathed thermocouples (MIMS-TCs), cable-type thermocouples or thermocouples with single sheet insulation is described. The use of so called fixed-point calibration rods makes it possible to calibrate such sensors at one particularly chosen temperature easily. A second sensor for measuring the calibration temperature (reference sensor) is not necessary. In most cases, the calibration procedure can be performed in exactly that situation where the measuring application normally takes place (in-situ calibration). Thus the whole individual measuring circuitry and its specific thermal conditions are involved during calibration process. Fixed-point calibration rods are useful for quality management inspections in rough industrial application areas, for outdoor surveying tasks as well as for precision temperature measurements in calibration baths or furnaces.
The paper presents structure and handling of fixed-point calibration rods. Examples of application including measuring uncertainty budgets are outlined.

Index Terms - MIMS thermocouples, slender temperature sensors, temperature calibration, miniature fixed-point cells, reproducibility, measuring uncertainty

1. INTRODUCTION

In the last decades miniature fixed-point cells have been developed and introduced into calibration laboratory practice. Different designs of such small sized crucibles are available: permanently built-in into industrial thermocouples or as exchangeable part of precision thermometers. Whether they are called “self-calibrating thermocouples” or “fixed-point thermocouples” - in any case they help to overcome the limitations of reproducibility of thermocouples [1] [2] [3].

With recently designed fixed-point calibration rods (FPCRs) a new construction is available permitting a convenient handling of miniature fixed-point cells and, in particular, an easy insertion of slender thermometers like MIMS-TCs[1], cable-type or other thermocouples with single-sheet insulation – see Fig. 1. They are often employed because of their small thermal time constants and their low thermal impact on the respective object to be measured.
However, the small cross sections of all these sensor types present some metrological disadvantages:
On the one hand, in the case of a finite input resistance of the measuring device, the relatively thin and, thus, high-impedance thermoelectric wires lead to a voltage drop which reduces the

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[1] MIMS-TCs: Mineral-insulated, metal sheathed thermocouples [4]. Due to their slender, bendable design they are extremely versatile. They are widely used in large quantities, for example in annealing processes concerning special-purpose steels (automobile and aircraft industry), for measurements on fast rotating component parts (turbine blades) or in places which are difficult to access

thermoelectric voltage to be measured. On the other hand, the small distances between the two parallel conductors involve low insulation resistances, thus leading to a reduction in the thermoelectric voltage, too, particularly in the case of higher temperatures.

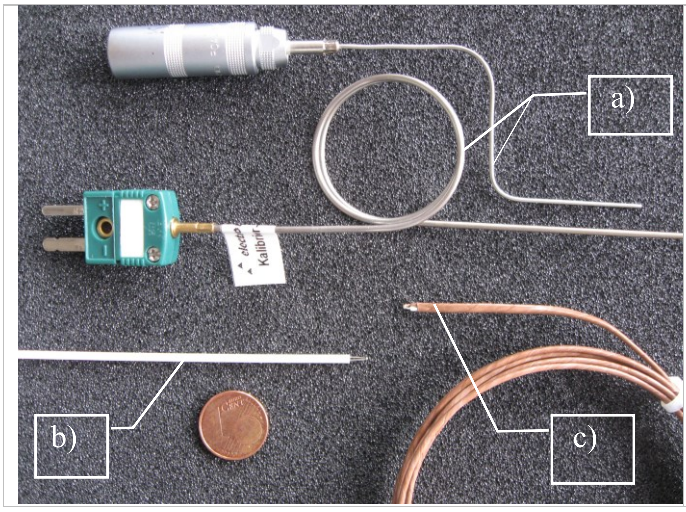

Figure 1
Examples of slender thermocouples, suitable to be calibrated with fixed-point calibration rods (FPCRs)
a) MIMS-TCs
b) TC probe with singe-sheet (ceramic) insulation
c) Cable type TC probes

By using FPCRs, such systematic errors can be recognized independently and consequently corrected in a process-related way. No additional reference thermometer or other expensive calibrated equipment will be required.

2. STRUCTURE OF FIXED-POINT CALIBRATION RODS

A FPCR as shown in Figure 2 consists of a stainless steel tube with a diameter of 6 mm. The tube length is selectable according to the insertion depth of the application. In the lower end, a miniature fixed-point cell is firmly installed. A funnel-shaped centering piece allows slender temperature sensors (diameter < *2 mm*) to be inserted in the fixed-point cell in a very convenient way. Inside this double-walled ceramic container, there is a substance whose melting point is exactly known (high-purity metal or alloy – see Table 1.

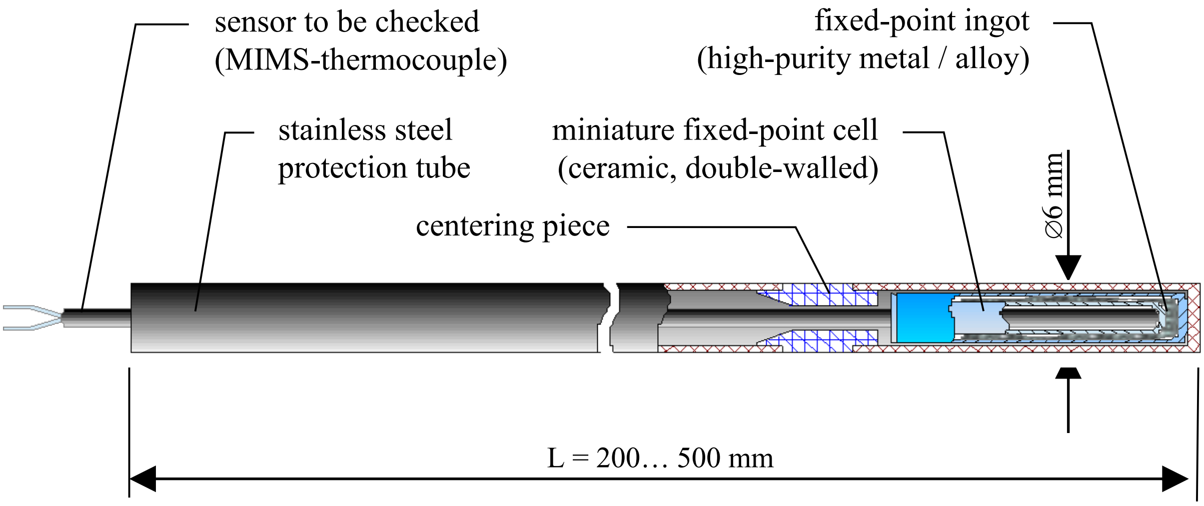

Figure 2 Schematic representation of a FPCR

Indium	156.60 °C	Gold-Indium	495.4 °C
Tin	231.93 °C	Aluminium-Copper	548.2 °C
Zinc	419.53 °C	Silver-Aluminium	567.8 °C
Aluminium	660.32 °C	Aluminium-Silicon	578.7 °C

Table 1
Various applicable high-purity materials and their melting or solidification temperature (fixed-points)

3. HOW IT WORKS

3.1 Brief overview

To calibrate temperature sensors normally a calibration sez-up is necessary comprising a calibration bath or furnace and an appropriate reference temperature measuring equipment. Then, the sensors have to be removed from the actual measuring application and built into the calibration device to perform a comparison at specifically chosen temperature points.

As opposed to calibration with FPCRs, the sensor can preferably remain in its application position or close to it. In any case, neither its specific measuring circuitry nor its environment needs to be changed, and a second thermometer for determining the reference temperature is not necessary either.

The calibration of a thermometer with the fixed-point rod includes the following four steps:

1. Installing the fixed-point calibration rod in a bath, a furnace or preferably - if it is a thermal controlled one - in the measuring process the sensor normally runs.

2. Inserting the slender thermometer to be calibrated in the rod and connecting it to the measuring device.

3. Heating up the whole assembly slowly until passing the melting point of the integrated fixed-point substance, observing or recording the temperature curve (see paragraph 3.2).

4. Analysing the temperature readings to find the holding period caused by the phase change transition of the fixed-point substance, and, determining the calibration point. There are at least 5 different ways to determine a high reproducible point within the melting period curve (see paragraph 3.3).

However, a correction value could be derived from difference between the displayed (more ore less incorrect) melting temperature T_{SP} and the well-known (true) melting point temperature T_{Fxp}. The difference dT_K is the correction value to be applied to the further measured temperature values ($T'_A = T_A - dT_K$). Figure 3 shows an example where the determination of determination on basis of a straight-line approximation.

3.2 Methods to generate evaluable melting plateaus

After the sensor is inserted in the FPCR, the whole assembly should be brought into a position where a controlled heating up passing the fixed-point temperature could be performed. This might be in a portable dry block calibrator, a calibration microbath, a well-fitting heater cartridge or another sort of small furnace. Some applications may even permit the installation of the FPCR with the sensor to be calibrated directly in the sensor's normal operation process, and the gradual heating up of the whole assembly in a controlled manner (annealing or flow-through furnaces). Although this would be the most effective and convenient situation, it is not necessarily found in each case.

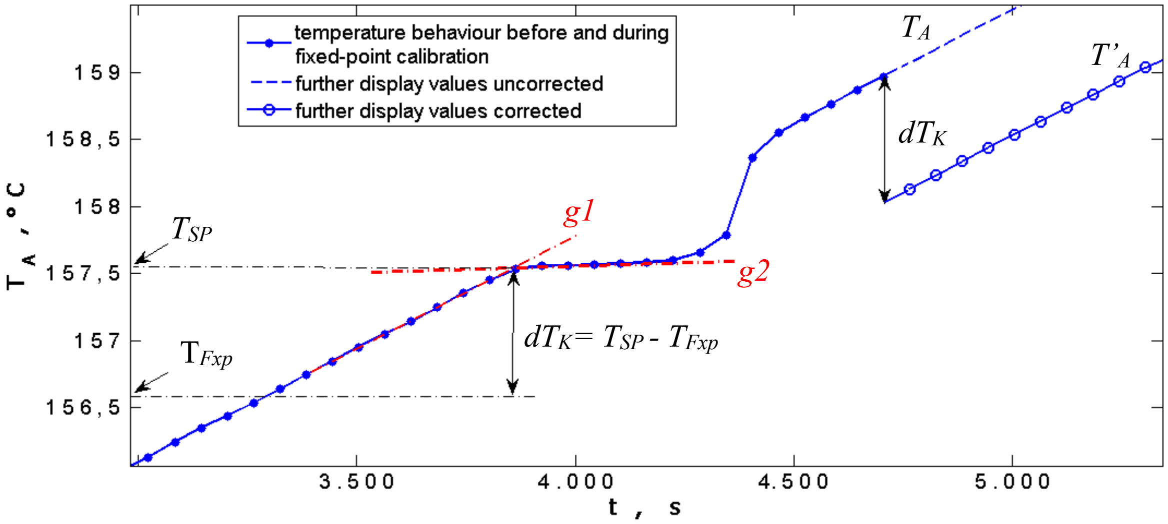

Figure 3 Recorded temperature curve when passing the melting point of the integrated fixed-point substance (Indium, m.p. 156.60°C). After determining the beginning of melting by the intersection point of straight lines *g1* and *g2*, a correction (here $dT_K = -0{,}95$K) can be derived and applied to temperature values measured further

To ensure a more or less controlled heating up process, the following methods have proved to be appropriate:

A. *Controlling a constant heating rate*
   This is a feature of many state-of-the art controllers. Heating rates of 0.1 to 2.0 K/min might be useful. A final maximal temperature should limit heating to about T_{Fxp} +10 K.

B. *Setting a fixed temperature value a few degrees above the melting temperature*
   The set temperature of the controller should be *0.5* to *5.0* degrees higher than T_{Fxp}.. Some experiments would be necessary to find an optimum between time consumption and accuracy of the result.

C. *Setting a constant heater power*
   Here, too, some experiments are necessary to find an appropriate setting. Any overheating should be limited to about T_{Fxp} +10 K.

D. *Shifting the assembly from a colder to a hotter region*
   This is similar to *b),* but needs some space and access to the assembly of sensor and FPCR. The hotter zone should have a temperature 0.5 to 5 K higher than T_{Fxp}. The colder zone should have a temperature at least *1* K lower than T_{Fxp}.

In general the starting temperature should be low enough to be sure that no melting has set in or also that a previous melting has been followed by a complete freezing process. The latter condition is not so easily fulfilled as one might expect. Especially high-purity metal melts can be supercooled to some degrees (2 to 20 K, sometimes 50 K !) before a freezing process is induced. On the other hand, the starting temperature should not be too low, especially if method *d)* is used and if high heating or cooling rates may cause thermal shocks and cracking of the ceramic crucible inside the FPCR.

All in all, depending on the fixed-point material used and on the heating-up method, some careful considerations and purposeful experiments with the applied equipment are recommended to be made in advance.

Here, it should be pointed out that the more slowly a continuous heating can be controlled, the closer the result of such a dynamic calibration will be to that of a static comparison calibration mentioned in Paragraph 3.1. However, due to technological requirements, a calibration run should never last longer than a few minutes. Hence, mean heating rates of about 0.5 to 2 K/min might be a good approach. Here, too, some prior experiments would help to find a good compromise.

3.3 Methods of determination of the fixed-point

However, when the process approaches the melting temperature of the fixed-point substance, a recording or at least attentive observing of the displayed temperature is necessary. Fig.4 shows some typical temperature curves recorded during the melting process.

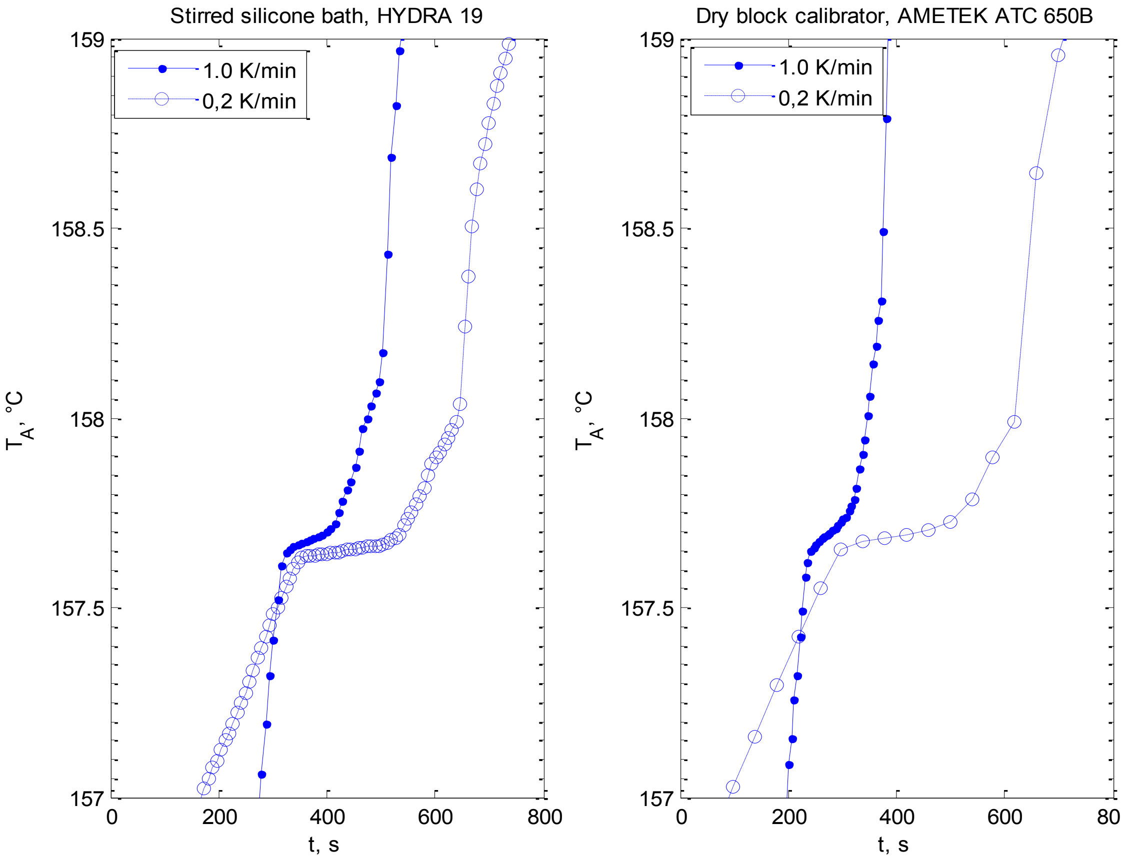

Figure 4 Typical temperature curves $T_A(t)$ recorded during continuous heating up of a silicone oil bath (left) and of a dry block calibrator (right).

An outstanding feature of all graphs is a nearly linear, more or less steep section, corresponding to the beginning of melting. This forms the part of the melting plateau which can best be evaluated. In general, the height and also the shape of the melting plateau varies slightly depending on operational parameters as heating rate and used ambient medium. In addition, some further influence factors were found: temperature profile, structure and type of the sensor, thermal contact between sensor and rod as well as between rod and ambient medium, etc. In all cases, however, a high repeatability of the melting section characteristic can be observed if the operational conditions are kept constant. Hence, the only challenge is to determine a calibration value which corresponds to the well-known ("true") melting temperature of the implemented fixed-point substance. Also for this task, different methods have been reported [5]. The four best suited approaches are demonstrated here by means of temperature values of a MIMS-TC applied in an Indium-FPCR recorded during heating up of *0.2 K/min* in a dry block calibrator (graph also included in Figure 3, on the right):

a) Approximation of the beginning of melting T_B as intersection point of straight lines g_1 (heat-up slope line), g_2 (plateau slope line) – see Figure 5

b) Inflection point T_I of the 3rdorder polynomial least square fit of the melting plateau section – see Figure 6

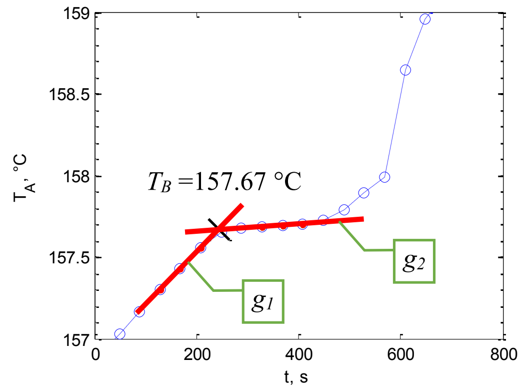

Figure 5 T_B – approximated begin of melting

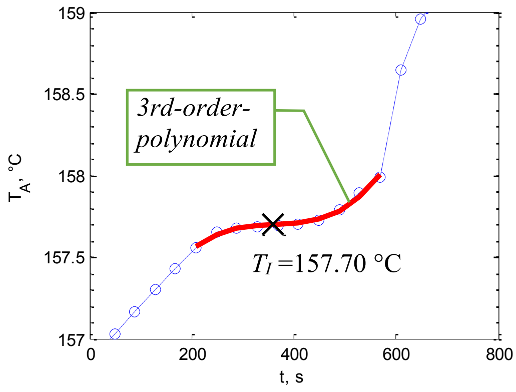

Figure 6 T_I – inflection point

c) Determination of the point of minimum plateau slope T_S - to be determined by calculation of 1st and 2nd derivative of the temperature records or similar – see Figure 7

d) Determination of the maximum of the temperature value distribution T_H (histogram) – see Figure 8. Depending on the e.m.f.-resolution and sample rate, an appropriate bin width w for building up the histogram is necessary, $w = 0.025$ to 0.1 K might be suitable.

Methods a) and b) require a recording and post analysis. If this is done by means of a PC, pre-filtering and plausibility test might be performed in addition to improve the resulting repeatability and reliability.

Methods c) and d) may even work without recording. A digital thermometer of a resolution of say 0.1 K and an attentive observation of the display would be sufficient to determine T_S or T_H (both probably not distinguishable). This would be interesting for in-situ field measurements or outdoor surveys. In case of recorded measurements made in analogy to *a)* and *b)*, post-processing improvements are possible here, too.

In Table 2, the results of the methods applied to all temperature curves presented in Figure 4 have been compiled.

Regardless of the method of determination applied, the results exemplify that the differences between T_B, T_I, T_S and T_H for each device set-up are some tenths of degrees maximum only. Their precision is much smaller than the tolerance of industrial thermocouples defined in normative standards [6]. So, each of the methods promise to be a robust and reliable calibration approach.

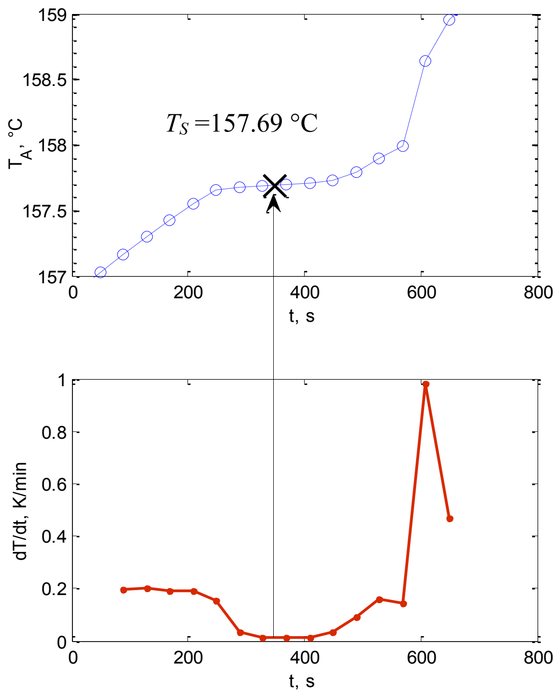

Figure 7 T_S – minimum of plateau slope

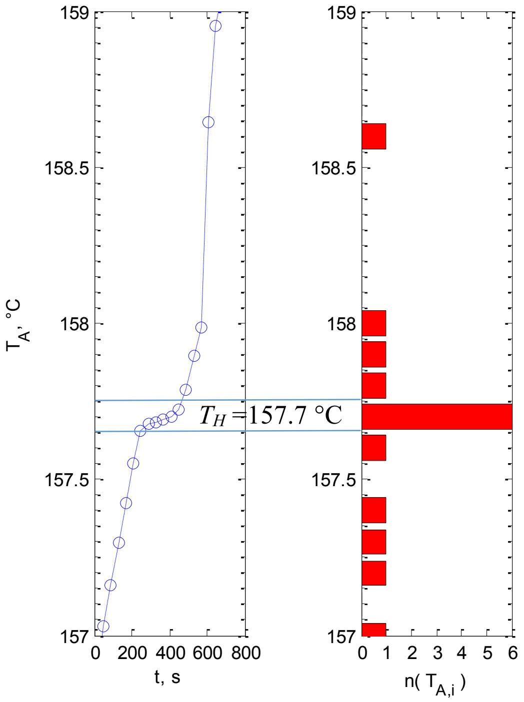

Figure 8 T_H - maximum of temperature value distribution (w = 0.1 K)

Method of generation of the melting plateau: *A)* Constant heating-up		**Method of determination**				**Averaged correction value** $dT_K=T_x-T_{Fxp}$, ($T_{Fxp,In}$=156.60°C) dT_K, **K**
Heating source	**Heating rate** dT/dt	***a)*** T_B, **°C**	***b)*** T_I, **°C**	***c)*** T_S, **°C**	***d)*** (w=0.1 K) T_H, **°C**	
Stirred silicone oil bath	1.0 K/min	157.65	157.67	157.69	157.7	+1.05 ± 0.04
	0.2 K/min	157.63	157.64	157.64	157.6	
Dry block calibrator	1.0 K/min	157.65	157.70	157.63	157.7	+1.08 ± 0.03
	0.2 K/min	157.67	157.70	157.69	157.7	

Table 2 Compilation of results of four test calibration set-ups – see Figure 4

4. ESTIMATION OF CALIBRATION UNCERTAINTY

To evaluate the reproducibility of the application of FPCRs the calibration uncertainty for two different exemplary device set-ups with the same MIMS-TC should be estimated. Basic set-up information is listed in Tab.3 .

	Laboratory conditions	**Industrial environment**
Ambient temperature	23 °C ± 2 K	25 °C ± 15 K
Sensor	MIMS-thermocouple type K, diameter 1.5 mm, length 1 m, extension wires; *e.m.f* = 22700 µV at 548.2 °C, *sensitivity:* 23.5 mK/µV at 548.2 °C [6]	
FPCR	ø6.0 mm, ingot AlCu (eutectic alloy, m.p. 548.2 °C ± 0.05 K*, calibrated with SPRT* , *k*=2)	
Cold-end-junction (CJ)	ice/water-mixture (0.0 °C ± 0.01 K*, estimated*)	miniature-size connector to handheld thermometer
Heating source	dry block calibrator (2 zones), fitting hole 6.3 mm diameter, heating rate controlled 0.5 K/min	annealing furnace with welded-in protecting tube, inner hole ø7.0 mm, heating rate: about 2.0 K/min
Measuring device	digital multimeter *HP3458A* 1) 100mV-range, 0.01µV-resolution, AutoCal, AutoZero, NPLC 100	handheld digital thermometer *LKM3000-K* 2) range 0-1300°C, 0.1 K resolution, internal CJ-correction (u_{CJC} = ± 0.5 K, after accommodation time of about 30 min, T_{amb} = 0…60°C, *experimentally measured*)
Sampling	rate 0.2 s^{-1}, PC-controlled and recorded	rate 1 sec^{-1}, no recording, observation of the display
Determination of fixed-point	straight-line approximation of begin of melting	observation of display, recognising the value with longest display time

Table 3 Basic calibration set-up information for two exemplary applications of FPCRs
1) Set-up according to "HP 3458A Multimeter. Calibration manual (Hewlett Packard)"
2) Set-up recommendations and uncertainty parameters acc. to manufacturer LKMelectronic GmbH

	Uncertainty Estimate	**Div.**	**Sensitivity coefficient**	**u_i mK**
Statistical standard uncertainty (repeatability under operational conditions, k =2)	5 µV	2	23.5 mK/µV	59
AlCu-m.p.temperature assignment (comparison calibration against SPRT, k =2)	50 mK	2	1	25
Drift of AlCu-m.p. temperature (long-term experience: < 0.3 K per 4 years [3])	25 mK	√3	1	14
Thermal field gradients (FPCR ↔ MIMS-TC ↔ heat source, estimation)	75 mK	√3	1	43
Inhomogeneity measuring circuit (fixed immersion, only short-time effects, estimation)	1 µV	√3	23.5 mK/µV	14
Cold-end junction (ice/water mixture)	10 mK	√3	1	6
Voltmeter drift (within 24h after ACAL)	0.4 µV	√3	23.5 mK/µV	5
Voltmeter linearity	0.1 µV	√3	23.5 mK/µV	1
Voltmeter resolution (+ noise)	0.5 µV	√3	23.5 mK/µV	7
Voltmeter temperature coefficient	0.2 µV	√3	23.5 mK/µV	3
Method of determination of fixed-point (repeatability with different person, k=2)	10 mK	2	1	5
Combined standard uncertainty $\sqrt{\sum u_i^2} = 81$ mK				

Table 4 Example of an uncertainty budget for a FPCR-application under laboratory conditions

Thus, the estimation of the expanded uncertainty for a laboratory calibration of a MIMS-TC by means of an AlCu-FPCR yields 0.16 K (k=2).

	Uncertainty Estimate	**Div.**	**Sensitivity coefficient**	**u_i mK**
Statistical standard uncertainty (repeatability under operational conditions, *k* =2)	100 mK	2	1	50
AlCu-m.p.temperature assignment (comparison calibration against SPRT, *k* =2)	50 mK	2	1	25
Drift of AlCu-m.p. temperature (long-term experience: < 0.3 K per 5 years [3])	25 mK	√3	1	14
Thermal field gradients (FPCR ↔ MIMS-TC ↔ heat source, estimation)	150 mK	√3	1	87
Inhomogeneity measuring circuit (fixed immersion, only short-time effects, estimation)	10 µV	√3	23.5 mK/µV	136
Cold-end junction (electronic CJC-correction)	500 mK	√3	1	289
DTM drift (only short-time effects, estimation)	100 mK	√3	1	58
DTM linearity	200 mK	√3	1	115
DTM resolution	100 mK	√3	1	58
DTM temperature coefficient (measurement)	200 mK	√3	1	115
Method of determination of fixed-point (repeatability, *k*=2)	200 mK	2	1	115
Combined standard uncertainty $\sqrt{\sum u_i^2} = 399$ mK				

Table 5 Example of an uncertainty budget for an industrial application of FPCR

So, the combined expanded uncertainty for a calibration of a MIMS-TC by means of an AlCu-FPCR (548.2 °C) in a rough industrial environment is estimated to be 0.8 K (*k*=2).

In comparison to the standard tolerance of type-K thermocouples (class 1: ± 2.2 K at 550 °C [6]) these results are apparently better. Regarding that standard tolerances are valid only in as-delivered-condition makes such small uncertainties, obtained in-situ, much more interesting.

Nevertheless, it should be underlined again that those first estimations are, strictly speaking, valid only for the moment and the thermal environment of the calibration process. To calculate the uncertainty of preceding measurements with the calibrated thermometer, additional contributions of errors have to come along (drift, thermal contact between TC and medium to measured, inhomogeneity of the TC, e.t.c.). In case of periodically repeated in-situ calibrations with FPCRs those additional errors too, could be minimized apparently.

5. SUMMARY

In the paper, the simple structure of recently developed slim fixed-point calibration rods as well as various applications have been presented. Due to their outer diameter of 6 mm, they enable an easy way of calibrating slender, fast-response temperature sensors.
Some exemplary calibration runs with MIMS thermocouples of a diameter of 1.5 mm show the high potential for improving the measuring accuracy near a well-defined temperature.
Uncertainty estimations for such fixed-point calibrations at a temperature of 548.2 °C (m.p. of AlCu eutect.) carried out in a laboratory or an industrial environment have yielded 0.2 K and 0.8 K, resp. (k=2). Although they are valid only temporarily and for specific calibration conditions, the results have proved to be a convenient solution to overcoming the limits of the reproducibility of standard thermocouples or other small-sized thermometers. In addition to laboratory applications, fixed-point calibration rods can preferably be used for outdoor surveying purposes and in rough industrial or hardly accessible areas.

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