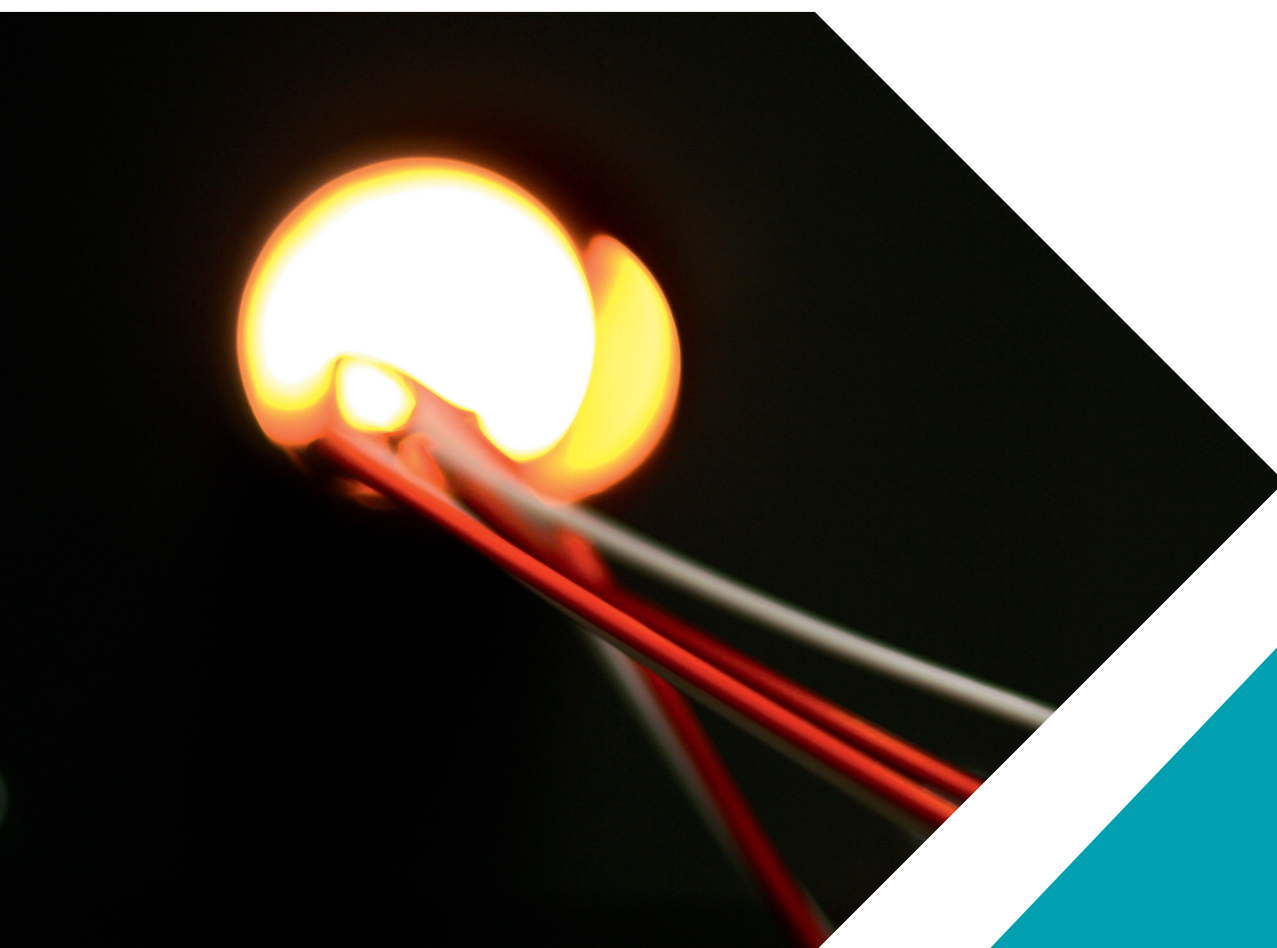


# Guidelines on the Calibration of Thermocouples

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Version 3.1 (02/2020)



**Thermometry**

## Authorship and Imprint

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# Guidelines on the Calibration of Thermocouples

## **Purpose**

This document has been produced to enhance the equivalence and mutual recognition of calibration results obtained by laboratories performing calibrations of thermocouples.

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## SCOPE

This guidance document has been written to meet the need for a basic advisory document for laboratories undertaking the calibration of thermocouples. It is valid primarily for thermocouple types standardised in accordance with temperature-*emf* reference tables adopted by the IEC as IEC 60584-1: 2013 [1] and IEC 62460: 2008 [2]. The Guide covers the temperature range -200 °C to +1600 °C, the calibrations being carried out in terms of the International Temperature Scale of 1990 (ITS-90) [3]. Although most of the topics covered may apply equally to 'non-standard' thermocouples, there may in these cases be other important considerations, outside the scope of these guidelines, that may have to be taken into account [4-6].

## 1 INTRODUCTION

- 1.1 A thermocouple consists of two dissimilar conductors connected together at the measuring junction (hot junction), the other ends (the reference junctions – cold junction) being connected, either directly or by some suitable means, to a device for measuring the thermo-electromotive force (*emf*, or voltage) generated in the circuit.
- 1.2 The electromotive force (*emf*) generated by a thermocouple is a function of the temperatures of the measuring (hot) and reference (cold) junctions but, more specifically, it is generated as a result of the temperature gradients which exist along the lengths of the conductors. Effective measurements and calibrations are possible only if the junctions are maintained in isothermal regions and at a depth sufficient to overcome heat losses (or gains), thereby ensuring that each junction effectively reaches the temperature of its environment.
- 1.3 The magnitude of the *emfs* depends on the materials of the conductors used for the thermocouple and their metallurgical condition. Subsequent changes in the material composition and condition caused by factors including oxidation, contamination, mechanical strain, or thermal shock, also influence and modify the *emf* and an associated calibration. Such change is influential only if it is located within the region of a temperature gradient. The change is not detectable by recalibration if, for example, a degraded length of conductor is located within the isothermal region of a calibration bath. Consequently, if during use of the thermocouple the part exposed to the gradient differs from the part exposed to the gradient during calibration, the calibration is not representative of the thermocouple behaviour during use.
- 1.4 The annealing of noble metal conductors, and their anneal state, have an important influence on their thermoelectric performance. This topic is covered in detail in [7].
- 1.5 With time and use, degradation of the thermocouple and its calibration is inevitable and in the longer term, therefore, a scheme of regular checks and eventual replacement should be established and maintained. For base-metal thermocouples used at high temperatures, replacement rather than recalibration is recommended.

## 2 INFLUENCES TO BE TAKEN INTO ACCOUNT

- 2.1 When the calibration is carried out, it should be ensured that effects due to the influences listed below are minimised. These influences should be taken into account for calculating the uncertainty of measurement stated in the calibration certificate.

## 2.2 Potential influences are:

- lack of immersion: poor contact and heat conduction along the thermocouple
- variation of temperature with time and spatial temperature distribution in the thermal source
- temperature variation in the cold (reference) junction
- parasitic thermovoltages, e.g. arising in connectors or from the use of a scanner or selector switch
- effects due to the use of extension or compensating cables
- electromagnetic interference
- mechanical stresses or deformations
- inhomogeneities along the lengths of the conductors due to
  - oxidation or other chemical contamination
  - changes in alloy composition, physical condition or crystal structure
  - mechanical stresses or deformation
- breakdown of insulation resistance.

These influences are discussed in the following sections.

## 3 EXTENSION AND COMPENSATING CABLES

**3.1** If, for practical reasons, the length of a thermocouple has to be increased this should be done using the correct extension or compensating cable. Extension cable consists of conductors made of nominally the same materials as the thermocouple conductors while compensating cable is made from a different pair of alloys. The cables are manufactured to match the *emf*/temperature characteristic of the thermocouple itself but over a restricted temperature range, no wider than -40 °C to +200 °C. Manufacturing tolerances are specified in EN IEC 60584-3 [8].

**3.2** These cables should preferably be connected permanently to the thermocouple. Alternatively, connections to thermocouple wires are often made using special plugs and sockets (also made of compensating alloys). It is important to ensure that these secondary junctions are not located in temperature-gradient regions, since they can then generate additional thermovoltage, and they should be shielded or insulated against draughts, radiation, and rapid changes in ambient temperature.

**3.3** The uncertainties of measurement associated with the use of extension and compensating leads are usually larger than those of continuous-wire thermocouples. This is attributable to the minor mismatch of materials and, in practice, difficulties in the measurement of the temperatures of the connections between conductors. The uncertainty of measurement may become similar to that of a continuous-wire thermocouple if the extension or compensating cable is included in the calibration. In this case, the extension or compensating cable is part of the thermocouple and should never be replaced by other wires even of the same type or batch. In order to estimate these uncertainty contributions, it is necessary to test the effect of changes in the temperature of the connections.

## 4 REFERENCE (COLD) JUNCTION

**4.1** Thermocouple temperature-*emf* tables have the ice-point, 0 °C, as the reference temperature and this traditional fixed point temperature is preferred for accurate and



reliable measurements. It is easily prepared using shaved or flaked ice mixed with water. De-ionised water is best, but in many countries tap-water may be good enough (with an ice point between  $-0,025\text{ }^{\circ}\text{C}$  and  $0\text{ }^{\circ}\text{C}$ ).

- 4.2 At the reference junction, commonly called the 'cold' junction, each thermocouple conductor is usually soft or hard soldered to a copper wire. Intermittent or permanent electrical failure at this connection can be caused by an oxide film forming on the thermocouple (base-metal) conductor or the copper wire. In preparation of the connection, the wire should be lightly cleaned with a fine abrasive paper. Each junction of wires should be insulated and the wires mounted in a light close-fitting sheath before insertion in ice/water baths. The copper wires should be taken from the same manufacturing batch.
- 4.3 Automatic cold-junction devices are used especially when large numbers and/or long-term thermocouple measurements are required. Their use should be accompanied by careful checks that the depth of immersion is adequate and that the total thermal loading does not exceed the capacity of the device. This may be achieved by monitoring the performance of one or two thermocouples used in the device, both with and without the full load of thermocouples, and comparisons can be made with their performance in an ice-bath. The cold-junction temperature should also be checked periodically.
- 4.4 The same remarks apply to reference junction boxes which may take the form of an insulated box containing reference junctions whose temperature is monitored by a thermometer either at ambient or a temperature provided by a thermostatically controlled heater. The effectiveness of the box's thermometer, thermal gradients along the reference junction boxes and controller should be checked periodically.
- 4.5 Cold-junction compensation is widely used in electronic temperature controllers and indicators. Electronic compensation modules are available, either mains or battery powered. It is important that the instruments are calibrated and used in environments where the temperature is not rapidly changing, and the effect of different environment temperatures should be checked [9].
- 4.6 If a reference (cold junction) temperature other than  $0\text{ }^{\circ}\text{C}$  is used with a thermocouple having a calibration referenced to  $0\text{ }^{\circ}\text{C}$ , the *emf* corresponding to the reference (cold junction) temperature chosen should be added to the measured *emf* output of the thermocouple. It is not correct to add the temperature of the reference (cold) junction as the correction.

## 5 INITIAL INSPECTION

Thermocouples are available in various forms of insulation and protective sheathing as well as in 'bare-wire' form. Initial inspection will therefore depend upon their construction and use. Obvious signs of mechanical defects, contamination, etc. should be recorded and the client informed if the laboratory feels that the validity or uncertainty of measurement in the calibration could be impaired. Any presence of moisture, particularly around compensating/extension lead connections, should be investigated as this may reduce the insulation resistance and/or lead to the generation of *emfs* by electrolytic action. Measurement of the insulation resistance is a convenient method to identify any moisture within the thermocouple.

## 6 HEAT TREATMENT

- 6.1 Every thermocouple which is to be calibrated should be homogeneous. Inhomogeneous thermocouples used under conditions different from which they were calibrated, especially different temperature gradients, will give erroneous results which could

amount to systematic deviations of several degrees Celsius, and in extreme cases considerably more (see Section 1.3).

- 6.2 Heat treatment or annealing of a thermocouple is intended to produce a uniform physical and chemical condition along the heated lengths of the thermocouple. It should be seen as a kind of adjustment and, in the case of recalibrations, such heat treatment should only be carried out with the formal agreement of the client.
- 6.3 For the best results, a thermocouple to be calibrated should first be annealed at maximum immersion at the highest temperature of intended use for several hours.

## 7 THERMAL SOURCES

- 7.1 Thermocouples are calibrated by measurement either at a series of fixed point temperatures, e.g. melting/freezing points, or by comparison with reference or standard thermometers, in thermally stabilised baths or furnaces suitable for the calibration, or by a combination of techniques, e.g. comparisons with a reference thermometer and/or fixed-point temperatures. Fixed-point(s) and standard thermometer(s) should be traceable to national standards. Generally, fixed point calibrations are only required for the calibration of noble metal thermocouples at the highest accuracy.
- 7.2 A thermally stabilised bath or furnace suitable for calibration is one in which spatial temperature profiling using two or more standard thermometers, at usually the mid-point and both ends of the working temperature range and within the working volume, has been shown to be within required limits. The inclusion of this profile in the calibration certificate may help resolve immersion problems, although the profile in furnaces can depend greatly on the dimension and sheath thermal characteristics of the thermocouple.
- 7.3 Temperature gradients within thermally stabilised baths or furnaces can be reduced or minimised by the insertion of an equalising block of high thermal conductivity and low specific heat, and drilled with thermowells of dimensions close to the thermocouple diameters to receive the standard and test instruments. Such a block is not always necessary, for example in multi-zone controlled furnaces and at high temperatures, where radiative heat transfer in a closed space is very efficient. Without a block, stabilisation may be achieved more quickly.
- 7.4 In liquid-filled baths, thermocouples should be loaded with a separation of about 1 cm and should not contact the enclosure bottom or sides which might be at a slightly different temperature from the liquid.
- 7.5 Standard and test thermocouples can be protected from contamination in a furnace or liquid-filled bath by inserting them in close-fitting thin-walled recrystallised alumina or quartz (up to 1000 °C) tubes with closed ends. However, longer immersion may be needed to compensate for the poorer thermal coupling and stem conduction effects.

## 8 IMMERSION DEPTH

- 8.1 When possible, thermocouples should be calibrated at the same immersion as required in normal use. However, thermocouples should be immersed to a depth sufficient to overcome heat losses or gains at high and low temperatures, respectively. Such effects are larger for large diameter wires and thick-walled insulators and sheaths. Where possible a thermocouple should be progressively immersed into a controlled calibration enclosure until further immersion shows no change in the measured *emfs*, indicating that an appropriate immersion depth has been reached. In some circumstances, sheaths and linings may need to be removed and a more suitable insulator substituted.

- 8.2** The considerations of Section 8.1 apply to both calibrations by comparison with a reference thermometer and fixed-point calibrations. A steady *emf* may be obtained, but this does not necessarily mean that the correct temperature has been reached. Adequate immersion is only demonstrated if the change in *emf* on withdrawing the thermocouple up to five centimetres is small compared with the required uncertainty of measurement in the calibration.

## **9 INHOMOGENEITY OF THE CONDUCTORS**

- 9.1** The *emf* of a thermocouple is produced in a temperature gradient and not at the thermocouple tip. In many cases the thermoelectric inhomogeneity of the conductors limits the measurement uncertainty.

- 9.2** Inhomogeneity can be quantified by moving the measuring junction in an environment with homogenous temperature distribution (e.g. a stirred liquid bath or a fixed point cell, or specialised single gradient scanner [12]). During the inhomogeneity test different parts of the wire are exposed to the region with the largest temperature gradient, resulting in changes of the *emf* if the thermocouple is not homogeneous. For calibration of reels of thermocouple wire, the inhomogeneity can be determined from the maximum variation of the calibration of two or more thermocouples taken from the reel.

- 9.3** It is recommended to estimate the uncertainty contribution from the inhomogeneity as rectangular contribution, with a full width equivalent to the largest difference found for any two measurements during the test. If the test was only performed over a small length of the thermocouple, the largest difference in *emf* found in the measurement should be taken as half width of the rectangular distribution. In cases where individual measurement of the inhomogeneity is not possible, it is recommended (for new thermocouples only) to take the following values as contribution ( $k = 1$ ) to the uncertainty [10]:

- Type K and N: 0,1 % of temperature in °C
- Type R and S: 0,02 % of temperature in °C
- Type B: 0,05 % of temperature in °C
- Au/Pt and Pt/Pd: 0,01 % of temperature in °C
- All other types: 0,25 % of temperature in °C

See Section 13 for guidance on the recalibration of used thermocouples.

- 9.4** For an estimation of the inhomogeneity at other temperatures than those tested, it may be assumed that for noble and pure metal thermocouples inhomogeneity can be expressed as a percentage of the total temperature, which is a sufficient approximation to the finding of [11]; although the published findings are expressed in terms of *emf*, here all advice is expressed in temperature terms to retain simplicity. The situation is very different for base metal thermocouples, which exhibit a wide range of temperature-dependent reversible and irreversible effects that make such an extrapolation impossible [13].

## **10 MEASUREMENT PROCEDURE**

- 10.1** Generally, thermocouples should be calibrated at decreasing temperatures, starting with the highest calibration temperature. Type K thermocouples, which are subject to calibration changes on temperature cycling to 500 °C or above, should be calibrated at

increasing temperatures, and the first calibration point repeated at the end as a check. The same considerations apply to a lesser extent to other base-metal thermocouples.

- 10.2** In fixed point measurements, it is prudent to measure the melting or freezing point of each realisation of temperature with a reference standard thermometer which should be dedicated for this purpose. An erroneous or false plateau can arise with the use of three-term temperature controllers or heat pipe furnaces which may hold the furnace very precisely near, but not at the fixed-point temperature. It is important, therefore, to witness the melting/freezing curve, and the undercool that precedes the temperature rise to the freezing point arrest.
- 10.3** In calibrations by comparison with a reference thermometer, it is advisable to use two standard thermometers which provide a cross check of one another and the calibration system. To reduce the effects of drift (instability) in the thermal source, one of the following measurement sequence should be followed:

$S_1, X_1, X_2 \dots X_n, S_2, S_2, X_n \dots X_2, X_1, S_1$

$S_1, X_1, S_1, X_2, S_1, \dots S_1, X_n, S_1, S_2, S_1$

where  $S_1$  and  $S_2$  are the two reference standards and  $X_1, X_2 \dots X_n$  are the thermocouples to be calibrated. Alternatively, if simultaneous measurement is possible the sequence can be:

$S_1 \& X_1, S_1 \& X_2, \dots S_1 \& X_n, S_1 \& S_2$

This sequence may be repeated to give four or more measurements on each instrument. The mean values are calculated and any corrections (for example, due to voltmeter calibration) are applied. The temperature is taken to be the mean value calculated from the results of  $S_1$  and  $S_2$ .

## 11 ELECTRICAL MEASUREMENTS

- 11.1** Electrical measurements are normally made using digital voltmeters or direct reading temperature indicators. All electrical measurement systems should be traceably calibrated over the whole of the required *emf*/temperature range.
- 11.2** Manual switchgear and dials on selector switches, reversing switches and manual potentiometers should be gently exercised on a daily basis through about twenty movements to clear oxide films and possible contact resistance.
- 11.3** When the closest accuracies are required measurements should be made of both forward and reverse polarities by means of a reversing switch. The average value of the measurements (ignoring the change in sign) eliminates or minimises the effect of the stray thermal *emfs* in the measuring system. Stray *emfs* can arise at any point in the measuring circuit where there is a change of temperature and at the juncture of dissimilar metals, e.g. copper wires and brass terminals. Suitable shielding and/or lagging and control of the ambient temperature should be provided. Digital voltmeters can behave differently in the positive and negative modes so both polarities should be calibrated if reversals are made. Alternatively the measuring circuit can be checked (and corrected) for any residual *emfs* by measurement of the circuit when the thermocouple is replaced by a short-circuit at the input connection terminals.

## 12 CHARACTERISTICS

- 12.1** Thermocouples are used to measure temperature in a certain range, not only at one temperature. The calibration laboratory therefore in many cases will provide the

customer with the characteristic of the thermocouple, i.e. an interpolation formula with a relation  $V = f(t)$ .

- 12.2** Thermocouples are standardised, and the reference function for the most common thermocouple types is defined in EN IEC 62460 and EN IEC 60584-1 [1,2]. The characteristic of individual thermocouples is usually close to the reference function. Therefore it is recommended to determine the deviation function  $g(t)$  from the reference function for the thermocouple under test, expressed as  $g(t) = (V - V_{\text{ref}})$ .
- 12.3** The deviation function  $g(t)$  usually is described as a low order polynomial. In many cases a second order (quadratic) deviation function is a good choice, but depending on temperature range, type of thermocouple and measurement uncertainty a linear deviation may be adequate, or a third order (cubic) deviation function may be preferable.
- 12.4** The coefficients of the deviation function should be determined using a least square fit procedure. The measurement temperatures should be approximately evenly spaced, and the number of points used for the fit should be at least two more than the number of coefficients to be determined.
- 12.5** The characteristic for the thermocouple under calibration is given by adding the deviation function to the reference function.
- 12.6** If it is within the calibration range, measurements at 0 °C should be made and included as a calibration point, in the same way as for all other temperatures.

## **13 RECALIBRATION**

- 13.1** There are no formally specified frequencies for the recalibration of thermocouples because their types, temperature ranges, construction, application, intensity of use are so numerous and varied. It should be expected that an in-house quality management scheme evolves a checking and recalibration programme to meet its requirements and experience.
- 13.2** Where there are long-term installations of thermocouples, calibration checks are best made *in situ* by providing for the insertion of a standard alongside the working thermocouple(s) as and when required. Alternatively, a thermocouple can be temporarily substituted for a standard thermocouple and their *emfs* compared. In practice, a programme of periodic replacement may be preferred.
- 13.3** A change in the *emfs* and calibration of a thermocouple as the result of use, or even as the immediate result of calibration, can be quantified by immersing the thermocouple in a thermally stabilised bath or furnace held at an appropriate temperature and measuring the output at a series of immersion depths spanning the normal working depth. If, finally, the thermocouple is substantially over-immersed, i.e. beyond any previous working depth, the measured *emfs* should closely approximate the value shown on the (first) calibration certificate at the corresponding temperature and corroborate the validity of the two (possibly different) calibration systems. Nevertheless, this effect of inhomogeneity (see Section 9) of the thermowires has to be taken into account when estimating the measurement uncertainty.
- 13.4** For base-metal thermocouples, a replacement with a calibrated thermocouple rather than a recalibration is often the best solution. Otherwise 'in-situ' calibration or checks are advised. Careful heat treatment can sometimes reduce inhomogeneity.

## **14 REPORTING RESULTS**

**14.1** The calibration certificate in which the results of the calibrations are reported should be set out with due regard to the ease of assimilation by the user to avoid the possibility of misuse or misunderstanding.

**14.2** The technical content of the certificate should comprise the following:

- a clear identification of the items subjected to measurement including the thermocouple(s), any compensating or extension cables especially when these are separate items and any other instruments (e.g. digital indicators) that form part of the whole measured system;
- the temperature range covered by the calibration;
- a statement of any heat treatment carried out before the calibration;
- the depth of immersion of the sensor, relative to the position of the main temperature gradient of the bath or furnace, together with a statement on the inhomogeneity of thermocouple;
- the measurement procedure used (e.g. 'fixed' points, comparison with reference thermometer(s)), increasing or decreasing calibration temperatures;
- any relevant environmental conditions;
- any standard or other specification relevant to the procedure used (e.g. IEC reference tables [1,2]);
- an evaluation of the uncertainty of measurement associated with the results.

## **15 UNCERTAINTY OF CALIBRATION**

**15.1** Uncertainties of measurement should be calculated in accordance with EA publication EA-4/02 'Expression of the Uncertainty of Measurement in Calibration' [13]. An example calibration showing likely sources of uncertainty is given in the appendix.

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## APPENDIX A

### Example of an evaluation of calibration results and an uncertainty budget

#### A1 Calibration of a type N thermocouple at 1000 °C

**A1.1** In this example, a Type N thermocouple is calibrated by comparison with two reference thermocouples of Type R in a horizontal furnace at a temperature of 1000 °C. The *emfs* generated by the thermocouples are measured with a digital microvoltmeter through a selector/reversing switch. All thermocouples have their reference junctions at 0 °C. The test thermocouple is connected to the reference point using compensating cables.

**A1.2** The temperature of the hot junction of the test thermocouple is given by:

$$\begin{aligned}
 t_X &= t_S(V) + \delta t_D + \delta t_F \\
 &= t_S(V_{IS} + \delta V_{IS1} + \delta V_{IS2} + \delta V_R - \frac{\delta t_{0S}}{C_{S0}}) + \delta t_D + \delta t_F \\
 &\cong t_S(V_{IS}) + C_S \cdot \delta V_{IS1} + C_S \cdot \delta V_{IS2} + C_S \cdot \delta V_R - \frac{C_S}{C_{S0}} \delta t_{0S} + \delta t_D + \delta t_F
 \end{aligned}
 \tag{A1.1}$$

The test thermocouple *emf*, with the cold junction at 0 °C, is given by:

$$V_X(t) \cong V_X(t_X) + \frac{\delta t}{C_X} - \frac{\delta t_{0X}}{C_{X0}} = V_{IX} + \delta V_{IX1} + \delta V_{IX2} + \delta V_{HX} + \delta V_R + \delta V_{LX} + \frac{\delta t}{C_X} - \frac{\delta t_{0X}}{C_{X0}}
 \tag{A1.2}$$

where

$t_S(V)$	temperature of the reference thermocouple as a function of <i>emf</i> with the cold junction at 0 °C. The function is given in the calibration certificate.
$V_{IS}, V_{IX}$	indications of the voltmeter (average of forward and reverse readings);
$\delta V_{IS1}, \delta V_{IX1}$	corrections due to the calibration of the voltmeter (average of forward and reverse readings);
$\delta V_{IS2}, \delta V_{IX2}$	corrections due to the limited resolution of the voltmeter (average of forward and reverse readings) – i.e. the addition or subtraction of an amount corresponding to half of the last digit of the voltmeter;
$\delta V_R$	correction due to parasitic <i>emfs</i> in the selector switch, and any other part of the measuring circuit not cancelled by the reversal of polarity;
$\delta t_{0S}, \delta t_{0X}$	temperature corrections due to the reference temperatures;
$C_S, C_X$	sensitivity coefficient of the thermocouples, in °C/μV, at the measuring temperature of 1000 °C;
$C_{S0}, C_{X0}$	sensitivity coefficient of the thermocouples, in °C/μV, at the reference temperature of 0 °C;
$\delta t_D$	drift of the reference thermocouples since the last calibration;
$\delta t_F$	temperature correction due to non-uniformity of the furnace;
$t$	temperature at which the test thermocouple is to be calibrated (calibration point);



- $\delta t = t - t_X$  deviation of the temperature of the calibration point from the temperature of the furnace;
- $\delta V_{LX}$  correction due to the compensation leads;
- $\delta V_{HX}$  correction due to inhomogeneity of the thermowires (if measured, or taken from this guide).

**A1.3** The reported result is the output *emf* of the test thermocouple at the required temperature *t*. Because the analysis consists of two steps — determination of the temperature of the furnace and determination of *emf* of the test thermocouple — the evaluation of the uncertainty of measurement is split in two parts. The standard uncertainty (coverage factor  $k = 1$ ) of each component is given in A1.14 and A1.15, evaluated as outlined below. The probability distributions for Type B components are assumed to be rectangular, and the estimated upper and lower limits of the uncertainties are therefore divided by  $2\sqrt{3}$ .

**A1.4 Reference standards:** The Type R reference thermocouples are supplied with calibration certificates that relate the temperature at their hot junctions to the *emf* produced, with their reference junctions at 0 °C. The expanded uncertainty of calibration in Table A1.14 at 1000 °C is  $U = 0,6$  °C (coverage factor  $k = 2$ ).

From previous calibrations, the drift of the values of the reference standards is estimated to be zero within the limits of  $\pm 0,3$  °C.

**A1.5 Sensitivity coefficients:** The sensitivity coefficients of the reference and test thermocouples have been taken from reference tables.

	1000 °C	0 °C
Reference thermocouple	$C_S = 0,077$ °C/ $\mu$ V	$C_{S0} = 0,189$ °C/ $\mu$ V
Test thermocouple	$C_X = 0,026$ °C/ $\mu$ V	$C_{S0} = 0,039$ °C/ $\mu$ V

In A1.14, the sensitivity coefficient for the uncertainty in the reference temperature of the standard thermocouples is  $C_S/C_{S0} = 0,077 / 0,189 = 0,407$ , see Equation A1.1

**A1.6 Resolution and calibration of the voltmeter:** A 4½ digit microvoltmeter has been used in its 10 mV range, resulting in resolution limits of 0,5  $\mu$ V at each indication. The voltmeter has been calibrated and respective corrections to the measured *emfs* are made to all results. The calibration certificate gives a constant expanded uncertainty of measurement of  $U = 2,0$   $\mu$ V for voltages below 50 mV (coverage factor  $k = 2$ ).

**A1.7 Parasitic voltages:** Residual parasitic offset *emfs* due to the switch contacts have been estimated to be zero within  $\pm 2$   $\mu$ V.

**A1.8 Reference temperatures:** The temperature of the reference point of each thermocouple is known to be 0 °C within  $\pm 0,1$  °C.

**A1.9 Non-uniformity of the furnace:** The temperature gradients inside the furnace have been measured by moving a thermocouple to different positions. At 1000 °C, deviations from non-uniformity of temperature in the region of measurement are estimated to be zero within  $\pm 1$  °C.

**A1.10 Compensation leads:** The compensation leads have been tested in the range 0 °C to 40 °C. *Emf* differences between the leads and the thermocouple wires are estimated to be less than 5  $\mu$ V.

**A.1.11 Inhomogeneity:** The thermocouples have been tested using a movable heating source. *Emf* differences due to inhomogeneities of the thermowires are estimated to be within  $\pm 15$   $\mu$ V, in Table A1.15.

**A1.12 Observations:** The voltmeter indications have been read in the following operational procedure, which gives 10 readings for every thermocouple and reduces the effects of temperature drift in the thermal source and of parasitic *emfs* in the measuring circuit:

1<sup>st</sup> cycle:

1<sup>st</sup> reference, test thermocouple, 2<sup>nd</sup> reference, 2<sup>nd</sup> reference, test thermocouple, 1<sup>st</sup> reference, and so on.

Reversed polarity.

2<sup>nd</sup> cycle:

1<sup>st</sup> reference, test thermocouple, 2<sup>nd</sup> reference, 2<sup>nd</sup> reference, test thermocouple, 1<sup>st</sup> reference, and so on.

The procedure requires the difference between the two reference standards not to exceed 0,3 °C. If the difference is not within these limits the observations have to be repeated and/or the reasons for such a large difference have to be investigated.

Thermocouple	1 <sup>st</sup> reference	Test	2 <sup>nd</sup> reference
Indicated voltage, after correction, see Equation A1.1	+10500 μV	+36245 μV	+10503 μV
	+10503 μV	+36248 μV	+10503 μV
	+10505 μV	+36244 μV	+10506 μV
	+10505 μV	+36249 μV	+10507 μV
	+10502 μV	+36253 μV	+10502 μV

	-10503 μV	-36248 μV	-10505 μV
	-10504 μV	-36251 μV	-10505 μV
	-10501 μV	-36254 μV	-10504 μV
	-10503 μV	-36244 μV	-10503 μV
	-10499 μV	-36244 μV	-10502 μV
Mean voltage	10502,5 μV	36248 μV	10504 μV
Standard deviation of the mean voltage $s(V)$	0,67 μV	1,26 μV	0,57 μV
Temperature of the hot junction	1000,473 °C ± 0,052 °C		1000,529 °C ± 0,044 °C
Temperature of the furnace	(1000,505 ± 0,034) °C		

**A1.13** The ten readings on each thermocouple are corrected, and one observation of the mean *emf* is deduced together with its standard deviation. The mean *emfs* of the reference thermocouples are converted to temperature observations using the temperature-*emf* relations given in their calibration certificates. By taking the weighted mean, they are combined into one observation of the temperature of the furnace at the location of the test thermocouple, assuming that  $\delta t_F = 0$ . The weighting factors for the calculation of the weighted mean are proportional to  $1/[s(V)]^2$ , with  $s(V)$  being the standard deviation of the mean *emf* of the thermocouples. The standard uncertainty of the furnace temperature has been calculated as the standard uncertainty of the weighted mean of the temperatures measured by the two thermocouples.

Note that this is only one (small) contribution to the uncertainty of the furnace temperature.

In a similar way, one observation of the *emf* of the test thermocouple is extracted.

#### A1.14 Uncertainty budget (temperature of the furnace):

Quantity	Symbol $X_i$	Estimate $x_i$	Standard Uncertainty $u(x_i)$	Probability Distribution	Sensitivity Coefficient $c_i$	Uncertainty Contribution $u_i(y)$
temperature of the furnace	$t_S$	1000,5 °C	0,01 °C	Normal	1,0	0,034 °C
voltmeter calibration	$\delta V_{S1}$	0 $\mu$ V	1,00 $\mu$ V	Normal	0,077 °C/ $\mu$ V	0,077 °C
voltmeter resolution	$\delta V_{S2}$	0 $\mu$ V	0,29 $\mu$ V	Rectangular	0,077 °C/ $\mu$ V	0,022 °C
parasitic emfs	$\delta V_R$	0 $\mu$ V	1,15 $\mu$ V	Rectangular	0,077 °C/ $\mu$ V	0,089 °C
reference temperature	$\delta t_{0S}$	0 °C	0,058 °C	Rectangular	-0,407	-0,024 °C
reference TC calibration	$\delta t_S$	0 °C	0,3 °C	Normal	1,0	0,3 °C
drift in reference thermocouples	$\delta t_D$	0 °C	0,173 °C	Rectangular	1,0	0,173 °C
furnace non-uniformity	$\delta t_F$	0 °C	0,577 °C	Rectangular	1,0	0,577 °C
	$t_X$	1000,5 °C				0,685 °C

#### A1.15 Uncertainty budget (*emf* of the thermocouple to be calibrated):

Quantity	Symbol $X_i$	Estimate $x_i$	Standard Uncertainty $u(x_i)$	Probability Distribution	Sensitivity Coefficient $c_i$	Uncertainty Contribution $u_i(y)$
test thermocouple <i>emf</i>	$V_X$	36 248 $\mu$ V	1,26 $\mu$ V	Normal	1,0	1,26 $\mu$ V
voltmeter calibration	$\delta V_{iX1}$	0 $\mu$ V	1,00 $\mu$ V	Normal	1,0	1,00 $\mu$ V
voltmeter resolution	$\delta V_{iX2}$	0 $\mu$ V	0,29 $\mu$ V	Rectangular	1,0	0,29 $\mu$ V
parasitic emfs	$\delta V_R$	0 $\mu$ V	1,15 $\mu$ V	Rectangular	1,0	1,15 $\mu$ V
compensation leads	$\delta V_{LX}$	0 $\mu$ V	2,9 $\mu$ V	Rectangular	1,0	2,9 $\mu$ V
temperature deviation of calibration point (see A1.14)	$\delta t_X$	0,5 °C	0,685 °C	Normal	38,5 $\mu$ V/°C	26,37 $\mu$ V
reference temperature	$\delta t_{0X}$	0 °C	0,058 °C	Rectangular	-25,6 $\mu$ V/°C	-1,48 $\mu$ V
inhomogeneity test TC	$\delta V_{HX}$	0 $\mu$ V	8,67 $\mu$ V	Rectangular	1,0	8,67 $\mu$ V
<b>Emf at 1000 °C</b>	$V_X$	36 229 $\mu$ V				28,02 $\mu$ V

### A1.16 Expanded uncertainties

The expanded uncertainty associated with the measurement of furnace temperature is

$$U = k \cdot u(t_X) = 2 \cdot 0,685 \text{ }^\circ\text{C} = 1,4 \text{ }^\circ\text{C}$$

The expanded uncertainty associated with the *emf* value of the test thermocouple at 1000 °C is

$$U = k \cdot u(V_X) = 2 \cdot 28,02 \text{ } \mu\text{V} = 56 \text{ } \mu\text{V}$$

### A1.17 Reported result

The Type N thermocouple shows, at the temperature of 1000,0 °C, with its cold junction at a temperature of 0 °C, an *emf* of 36 230  $\mu\text{V} \pm 56 \mu\text{V}$ .

The reported expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor *k* such that the coverage probability corresponds to approximately 95 %.

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