Good practice guide containing experimental results and recommendations for the selection, preparation and calibration of the temperature sensors

1. Scope

The purpose of this Guide is to outline the basic methodologies for estimating the influences of real condition to the thermocouples behaviour. It is intended for those laboratories undertaking the selection, preparation and calibration of the temperature sensors to be used in high temperature thermal conductivity measurement devices. It has not the aim to replace or to harmonize the existing standards.

2. Introduction

This document will establish a procedure for proper selection and use of the thermocouples in HTGHP applications. High temperature measurements bring high demands for temperature sensors to obtain acceptable precision, sensitivity and long-term stability. Temperature sensors intended to be used in high temperature guarded hot plate (HTGHP) apparatus, except for these requirements, should meet additional criteria. They need to be as thin as possible to minimize disruption of desired temperature profile inside a specimen and avoid significant heat losses. Regarding the temperature in HTGHP apparatus is monitored at various positions e.g. to check uniformity of temperature on plate surfaces, the temperature sensors used should fulfill high level of batch agreement to behave comparably in the course of measurements. On the other hand, the condition of batch agreement is not substantial when the temperature sensors can be regularly and properly calibrated. Therefore, they should be removable for the calibration additionally.

The most important standards and technical guidelines are outlined below

- EN (IEC) 60584-1: 2013. Thermocouples, Part 1, EMF specifications and tolerances
- Calibration Guide, EURAMET cg-8, CALIBRATION OF THERMOCOUPLES, Version 2.1 (10/2011)
- Deliverable 1.3.1 - Report on the survey about the thin temperature sensors, SIB 52 Thermo, Metrology for thermal protection materials
- Deliverable 1.3.3 - Report on investigation of the reliability of thin thermocouple temperature sensors for use in HTGHP, SIB 52 Thermo, Metrology for thermal protection materials
- Deliverable 1.3.5 - Study of the electrical insulation and long-term stability of response of 3 types of temperature sensors for HTGHP, SIB 52 Thermo, Metrology for thermal protection materials

3. Selection of thermocouples for temperature measurements in HTGHP

Among standardized base metal thermocouples type K and type N thermocouples correspond to the operating temperature range of HTGHP (upper measurements temperature 850 °C). At high temperatures they are most oxidation-resistant base metal thermocouples. Type K composed of nickel-chromium alloy and nickel-aluminum alloy legs is widely used as general purpose thermocouple with appreciable high sensitivity. Although type K thermocouple is recommended to be used up to 1100 °C, both its elements are subject to oxidation already at lower temperatures. Type N thermocouple consisting of nickel-chromium-silicon alloy and nickel-silicon alloy moieties compared to type K offers enhanced thermoelectric stability higher reproducibility, and higher accuracy at similar sensitivity level. More favorable characteristics (except the sensitivity) at high temperatures can be achieved using noble metal containing thermocouples which are often used as reference thermometers for calibrations:

standardized type R and type S thermocouples (Pt/PtRh) or even better, due to undesirable oxidation of Rhodium at high temperatures, thermocouples composed of pure oxidation-resistant elements e.g. Au/Pt or Pt/Pd. Nevertheless, their thermoelectric qualities are accompanied by a high costs.

Mineral insulated metal sheathed thermocouples
Regarding the use in the HTGHP, the construction of thermocouple sensor should enable following features keeping its diameter as small as possible:

- Sufficient thermoelectric sensitivity
- Sufficient electrical insulation of thermocouple legs
- Isolation of thermocouple wires to prevent the contact with environment that may cause considerable deterioration and thus influence the long-term stability
- Good resistance to thermal-shock

It seems that mineral-insulated metal-sheathed thermocouples (MIMS) are the only choice which can challenge these requirements at small sensor diameter. In MIMS construction, thermocouple wires are insulated by refractory oxides, (typically magnesium oxide) and sheathed by integral layer of metal. MIMS design allows manufacturing of thermocouple cables that can be bent and thus adapted to desired profile.
MIMS thermocouple long-term stability depends on many factors. Except for thermocouple type, sheath and mineral insulation materials can influence sensor quality remarkably. Materials used should not contaminate the thermocouple elements, as a consequence high purity materials for insulation are required (e.g. platinum is susceptible to contamination by many elements: Si, Al, P, In, S, Ni…). Also the sheath diameter which is generally positively correlated with the thermocouple long-term stability is important variable.

Recommendation
On the basis of a literature survey about thin temperature sensors intended for use in HTGHP, MIMS thermocouples seem to fulfill desired criteria as an only candidate. Type N thermocouples sheathed by advanced materials and R/S type thermocouples were proposed for using on this applications. Comparing type N and type R or type S thermocouples, the use of type N thermocouple is more favorable due to considerably higher thermoelectric sensitivity.

As it has been shown, the outer diameter of thermocouple sensor has a significant influence on the thermocouple long-time drift, therefore a compromise between temperature sensor thickness and desired sensor stability has to be found. This can be done be carefuly selecting of the thermocouples with good batch agreement.

4. Reliability of thermocouple sensors

For investigating the suitability of different thermocouples, there several tests were performed. Users could rely on these results, or can make their own measurements according to below described scheme with their batch of thermocouples.

Stability measurements in air furnace
There were two batches of measurements performed. Totally 7 N-type MIMS thermocouples with marking LNE1, LNE2, LNE3, LNE4, NPL1, NPL2, and NPL3 were investigated within 1st batch. After

annealing and homogeneity measurements in oil bath at 200 °C, thermocouples were tested for thermoelectric voltage drift in horizontal air furnace Heraeus at nominal temperatures 600 °C and 815 °C, respectively. Reference junction was realized using isothermal cold junction box which temperature was monitored by Pt100 resistance thermometer. During the measurements, the temperature inside air furnace was checked periodically with calibrated reference type S thermocouple. To minimize the effect of furnace drift, thermocouple readings were compared to those of marked as LNE1 at nominal temperature 815 °C. As can be seen from fig. 1, voltage of thermocouples did not drift significantly within 250 hours (or all thermocouples drifted in the same fashion). The deviations from initial values were lower than 10 µV (~ 0.26 °C) for individual thermocouples.

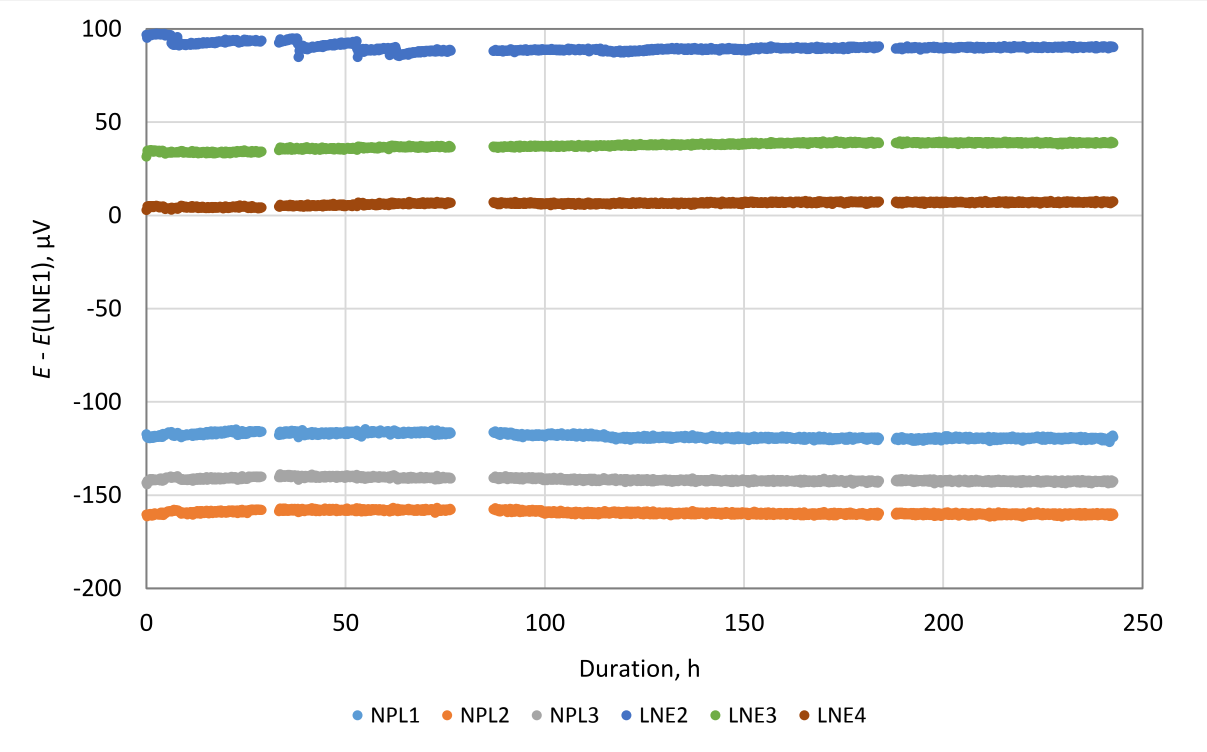

Figure 1. Difference of thermoelectric voltage from thermocouple LNE1 at nominal temperature 815 °C

Within 2nd batch, 9 MIMS thermocouples were investigated in total. 3 type S sheathed thermocouples (sheath diameter 1.5 mm) and 6 type N thermocouples (sheath diameter 1 mm) were placed in air furnace together with reference type S thermocouple at nominal temperature 825 °C. Contrary to measurements carried out in 1st batch, the reference thermocouple was placed in the furnace for the whole duration of measurements. Before and after the measurements in air furnace, reference thermocouple was calibrated in fixed points (Zn, Al, Ag) to verify its stability. The temperature deviations from reference thermocouple are presented in fig. 2. For the majority of investigated thermocouples, the temperature drift was better than 1 °C for the whole measurement duration.

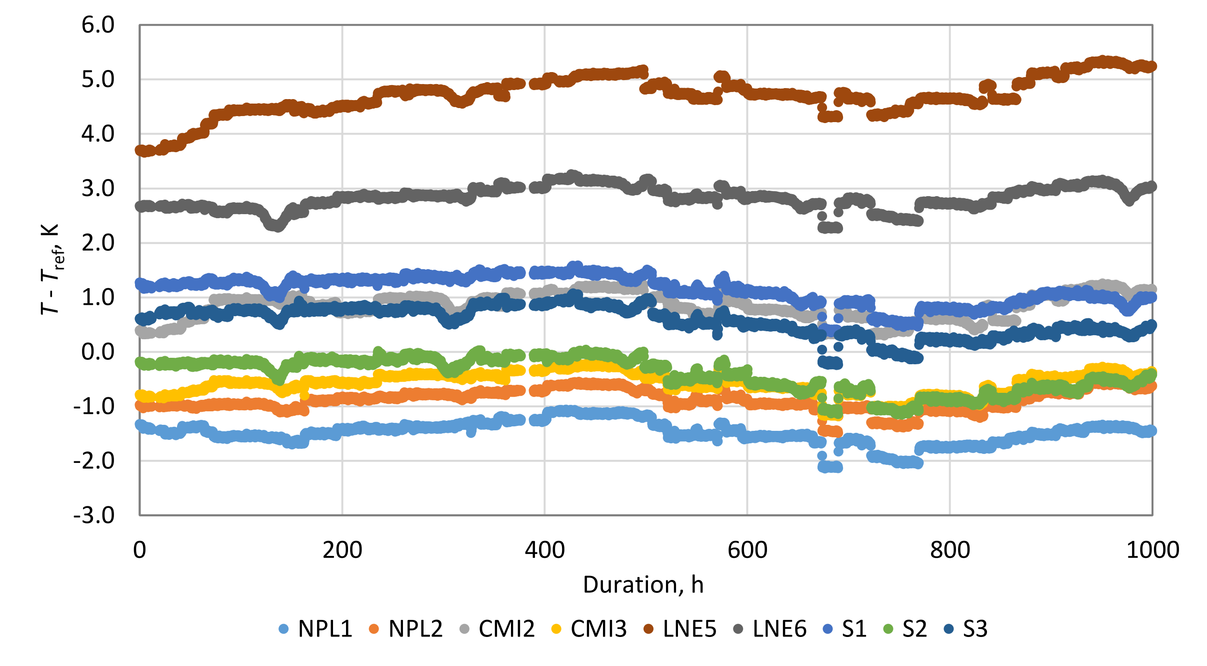

Figure 2. Temperature difference from temperature measured by reference thermocouple at nominal temperature 825 °C

Homogeneity measurements in oil bath at 200 °C

Temperature homogeneity measurements were carried out in oil bath at nominal temperature 200 °C for all investigated thermocouples. There are significant influence of high temperature treatment for both homogeneity and drift of the thermocouples. The drift of temperatures calculated according to EN 60584-1 from respective thermoelectric voltages is within 1 °C for all thermocouples.

The typical results of homogeneity measurements are shown in figures 3 and 4 as a temperature deviation with respect to full immersion of individual thermocouples. For both batches, the exposure of thermocouples to temperature above 800 °C in air furnace for a longer period of time led to worsen their homogeneity. Probably, this indicates a change of thermocouple alloy local composition. In most pronounced case, the absolute temperature difference from full immersion attained 1 °C.

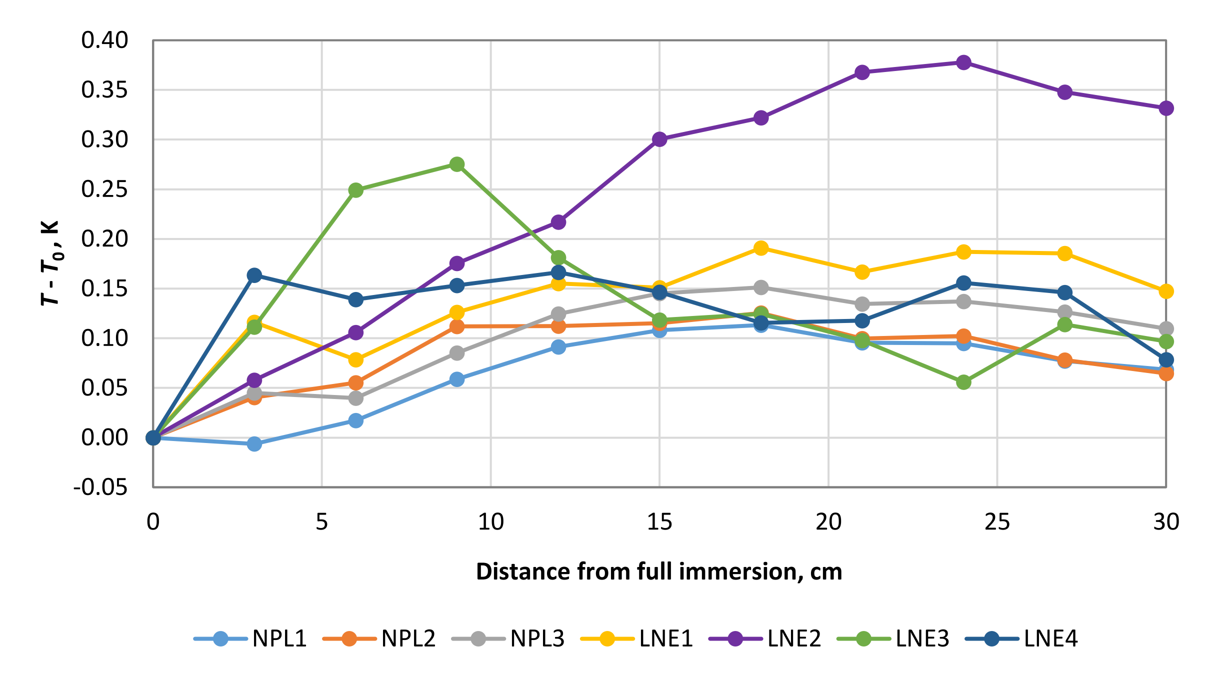

Figure 3. Temperature difference related to full immersion of thermocouples in oil bath at nominal temperature 200 °C, 1st batch, before high-temperature measurements

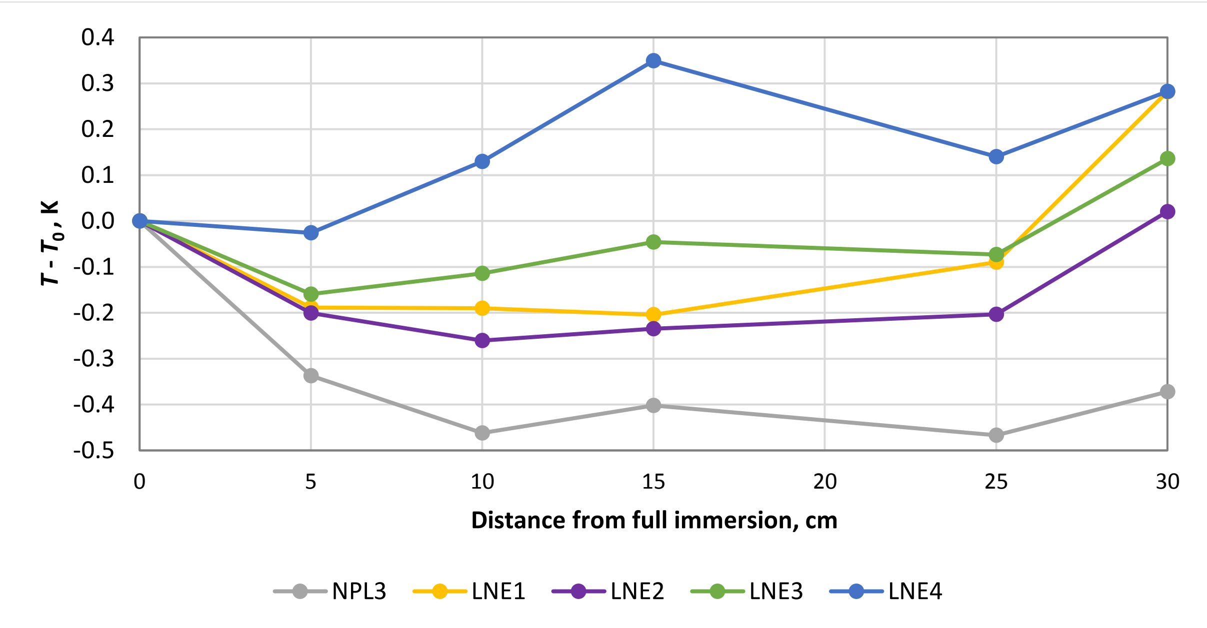

Figure 4. Temperature difference related to full immersion of thermocouples in oil bath at nominal temperature 200 °C, 1st batch, after high-temperature measurements

5. Calibration of thermocouples

The common practise is to use thermocouples from one batch and select that pc, which have similar behaviour. This minimalised the overall uncertainty of difference measurements. Calibration in laboratory covered by accreditation or CIPM MRA logo is generally required to fulfil uncertainty of thermocouples. In usage of HTGHP there are three types of thermocouples.

1. Thermocouples used for difference measurements – the most important requirement is similar behaviour (batch agreement) and similar measurement condition (e.g. immersion)
2. Thermocouples used for temperature control – here mainly good overview about drift is important, accuracy requirements has minor influence.
3. Thermocouples used for balance control – here the proper evaluation is needed.

Proper evaluation of measurement uncertainty included calibration of all used equipments, homogeneity and drift of thermocouples and thermal condition of thermocouple in real condition. The estimation of homogeneity and drift is possible from above measurements. The influence of real condition could be estimated from calibration in different immersion or from calibration IN-SITU. Both possibilities not fully agree with real condition and should be used as an estimation only. The calibration in real condition (with measured material inside) will have big uncertainty because of temperature profile inside specimen.

The possibility of calibration in different immersion will increase the overall uncertainty of calibration. Typical result for calibration in liquid bath is shown at following picture.

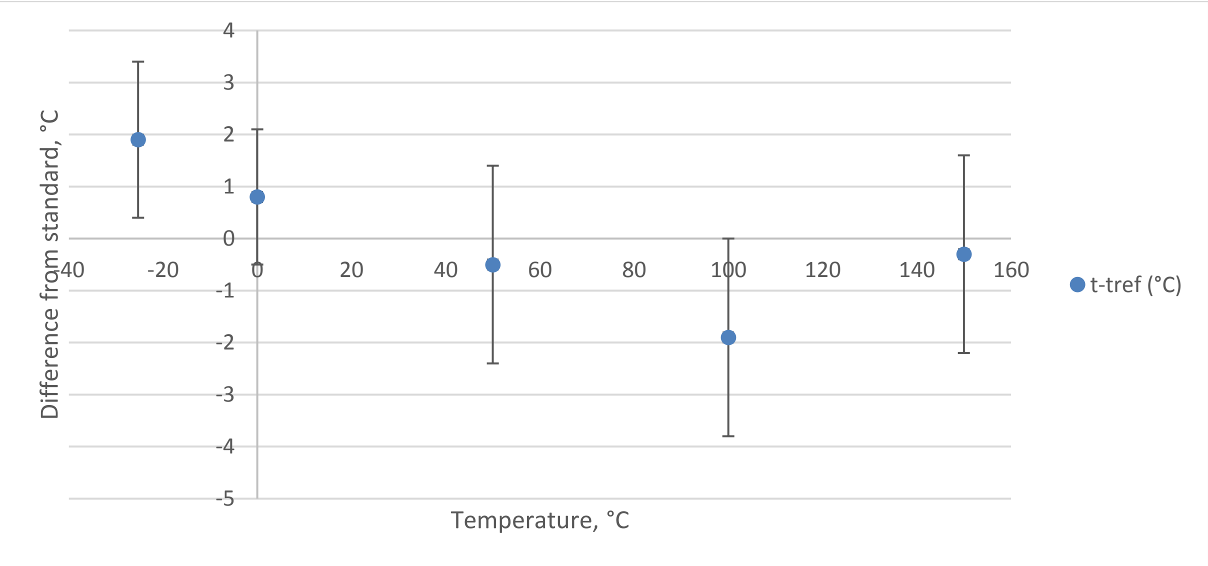

Figure 5. Typical result of thermocouple calibration in liquid bath at immersion of 34 mm.

The influence of immersion for different thermocouple type is shown on following picture.

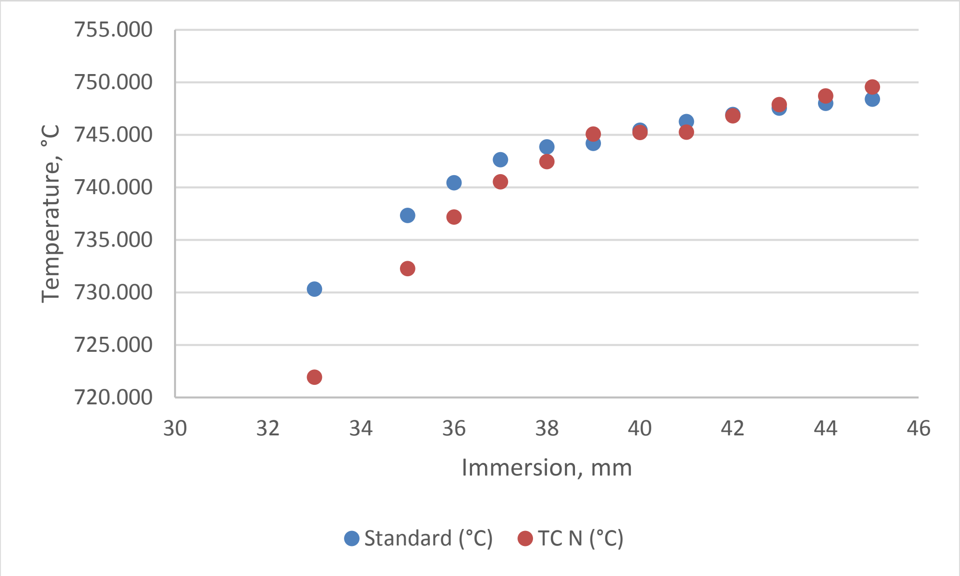

Figure 6. Typical result different immersion measurement.

6. Uncertainty of thermocouple calibration

Uncertainties of measurement shall be calculated in accordance with EA publication EA-4/02 'Expression of the Uncertainty of Measurement in Calibration'

To maintain high reliability of temperature measurement using thermocouples, their periodical calibration is needed. Following the EURAMET calibration guide for thermocouples, there can be identified various sources affecting the final calibration uncertainty (and also own temperature measurement):

- poor contact or 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
- effects due to the use of extension or compensating cables
- electromagnetic interference
- mechanical stresses or deformations
- inhomogeneities
- oxidation or other chemical contamination
- changes in alloy composition, physical condition or crystal structure
- breakdown of insulation resistance

When the calibration at a fixed point is considered, the electromotive force (emf) $E_X(t_X)$ of the thermocouple indicated by voltmeter can be expressed as

$$E_X(t_X) = E_X(t_F) + (\delta t_F + \delta t_{DF} + \delta t_{HF}) \cdot C_{FX} + \delta E_{X1} + \delta E_{X2} + \delta E_{X3} + \delta E_{X4} + \\ + \delta t_{0X} \cdot C_{0X} + \delta E_{\mathrm{Hom}} \cdot E_X(t_X) / E_{\mathrm{Ag}} \qquad (1)$$

where $E_X(t_F)$ is emf at the fixed point temperature t_F, δt_F correction on basis of the calibration of the fixed point, δt_{DF} correction on basis of the drift of the fixed point temperature, δt_{HF} correction on basis of a heat flux along the thermocouple, C_{FX} sensitivity of the thermocouple at the fixed point temperature, δE_{X1} correction on basis of the voltmeter calibration, δE_{X2} correction on basis of the voltmeter resolution, δE_{X3} correction on basis of the voltmeter drift, δE_{X4} correction due to the influence on ambient parameters and connection leads, δt_{0X} temperature correction due to the reference temperature, C_{0X} sensitivity of the thermocouple at the reference temperature of 0 °C, δE_{Hom} correction due to the influence of inhomogeneous thermocouple wires at the temperature of fixed point, and E_{Ag} is emf at the freezing point of silver.

7. Conclusion

The purpose of this Guide is to outline the basic recommendation for methods for those laboratories undertaking the selection, preparation and calibration of the temperature sensors to be used in high temperature thermal conductivity measurement devices. The sources of uncertainty for real condition measurement are presented.