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Procedure for an Investigation of Drift Behavior of Noble Metal Thermocouples at High Temperature

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1 Procedure for an Investigation of Drift Behavior of Noble 2 Metal Thermocouples at High Temperature

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6 Abstract

7 This paper presents the effects of short-term and long-term temperature exposure
8 on noble metal thermocouples in the range from 1000 °C to 1720 °C in oxidizing
9 atmosphere (air). As thermocouples voltage output depends on the wire material
10 of which the sensors are constructed, high-temperature and long continuous expo-
11 sure to limit temperatures can introduce changes to the materials composition and
12 structure. This can result in drift of generated voltage independent of the thermal
13 environment and into reduced lifetime of the sensors. The intensions of conducted
14 measurements were to determine the drift and lifetime of commonly available
15 alumina-sheathed noble metal thermocouples and furthermore to establish trace-
16 able techniques to enable lifetime testing and thermoelectric stability evaluation of
17 noble metal thermocouples at high temperatures. Result presented within this paper
18 is not intended to test the capabilities of thermocouple Types B, R and S in gen-
19 eral, but to show a possible behavior in the above-specified temperature conditions.
20 This work puts more emphasis on the procedures that can be used for regular checks
21 of intensely used thermocouple. Results obtained by this study show that the long-
22 term temperature drift of Type B thermocouples at 1600 °C, and R-, S-type ther-
23 mocouples at 1000 °C are much smaller than thermoelectric stability declared by
24 IEC 60584-1:2013 standard (International standard IEC 60584-1:2013, Thermocou-
25 ples—Part 1: EMF specifications and tolerances, 2013) tolerance classes after expo-
26 sure to thermal stress up to 4 months. More specifically tolerance for class 1 ther-
27 mocouple Types R, S from 0 °C up to 1100 °C is ± 1 °C and for class 2 tolerance
28 for Type B at 1600 °C is ± 1.5 °C. The short-term thermoelectric stability of R- and
29 S-type thermocouples exposed to 1600 °C for repeated 8 h periods has been within
30 2 °C when measured by comparison with a reference Pt–Pd thermocouple at 960 °C.
31 The short-term thermoelectric stability of B-type thermocouple when exposed to
32 1720 °C for repetition of 8 h has been within 1 °C.

33 **Keywords** Drift testing · High temperatures · Long-term stability · Noble metal ·
34 Thermoelectric stability · Thermocouple

35 1 Introduction

36 Noble metal thermocouples were designed to perform precise measurement in
37 laboratory and industrial applications with high precision, long lifetime and mini-
38 mum drift. These thermocouples are superior in many ways to base metal ther-
39 mocouples, but they are still affected by various influences. The most significant
40 changes in the thermocouple-generated voltage (emf) are caused by altering the
41 composition of the lead material. For example, the 0.1 % addition of Fe, Cr, Si,
42 Ni, Rh or Cu to the pure Pt wire that is used in many noble metal thermocouples
43 creates a change of Seebeck coefficient for the temperature range from 400 °C to
44 1100 °C by 1.7, 0.75, 0.64, 0.33, 0.21 or 0.15 μVK^{-1} , respectively [1]. As can be
45 seen, the effect of contamination on the Seebeck coefficient is considerable and
46 therefore these thermocouples should not be used in reducing oxidizing atmos-
47 phere or in the presence of metallic vapors. Low oxygen partial pressure and vari-
48 ous refractory oxides like Al_2O_3 have an effect on the noble metal thermocouples
49 as well [2]. Noble metal thermocouples are used in industrial conditions for long-
50 term temperature monitoring and regulation. By this long time exposure to tem-
51 perature and contaminated environment, a continuous change of the thermocou-
52 ple output independent of the thermal environment may occur. This divergence
53 due to changes within the thermocouple lead material is referred to as drift. In
54 a majority of measurement applications, the thermoelements are well protected
55 from outside contamination and therefore the effects that were mentioned ear-
56 lier are minimized. Beside the above-mentioned mechanisms, further effects that
57 cause the change within the Seebeck coefficient and subsequently in generated
58 electromotive force (emf) are mainly caused in Type R and S thermocouples by
59 grain growth and embrittlement in the pure platinum thermoelement after a long
60 exposure to temperatures above 1100 °C [3, 4, 13]. Furthermore, the high mobil-
61 ity of possible contaminants at temperatures above 1500 °C together with long
62 exposure times affects the Seebeck coefficient of Type B thermocouples [4].

AQ1

63 Due to the nature of their use and high demand for more reliable and more
64 precise measurements of temperature, it is necessary to examine the performance
65 of these sensors in long-term application. Although the drift and thermoelectric
66 stability measurements were performed in the past for noble metal [1, 2, 11] and
67 base metal thermocouples [6], the used techniques vary and the results are rarely
68 published. Furthermore, the available results are not up-to-date, and the previ-
69 ously stated performances may have changed due to the developments and inno-
70 vation in thermocouple manufacturing. This study which is linked to the EMRP
71 Joint Research Project “HiTeMS” (high-temperature metrology for industrial
72 applications (> 1000 °C)) was undertaken by two national metrological institutes
73 (NMI) CMI (CZ) and TUBITAK UME (TR). It deals with drift problematic in
74 noble metal thermocouples. The goal of this paper is to present the drift and ther-
75 moelectric stability results obtained by the different NMI for Type S, Type B and
76 Type R thermocouples. The study was performed on a total of ten noble metal
77 thermocouples (3× Type S, 4× Type B and 3× Type R) which were distributed
78 to the partners for performing short- and long-term drift testing the temperature

79 range from 1000 °C to 1720 °C. Thermoelectric stability measurements using
80 fixed-point cells after the aging period were applied to all thermocouples with
81 the exception of two R type and two S type due to occurrence of bending. This
82 process is described in the following sections together with the results of thermo-
83 electric stability of the tested thermocouples.

84 Tested thermocouples were 700 mm to 800 mm long and were housed in an alu-
85 mina sheath with an outer diameter of 6 mm with a wall thickness of 1 mm. During
86 the CMI measurements, a 10-year-old thermocouple Type B was also used for long-
87 term drift test from local Czech manufacturer.

88 2 Experimental Facilities

89 2.1 CMI Facilities

90 CMI has tested four Type B, one Type R and one Type S thermocouples. These
91 thermocouples were commercially made with an outer and inner insulation made of
92 high-purity alumina (99.7 %), and the total length of the tested thermocouples outer
93 insulation was 600 mm for Type R and Type S and 700 mm for Type B thermocou-
94 ples. The outer diameters of the sensors were 6 mm for the Type B and 7 mm for
95 Types R and S with a wall thickness of 1 mm. The reference junction was housed in a
96 metal-sheathed capsule.

97 A three-zone horizontal furnace with sufficient homogeneity and thermal stability
98 was used to perform the short- and long-term measurements. The furnace heating
99 elements were constructed for continuous usage at 1800 °C. The thermal stability
100 of the CLASIC5018T3Z furnace at 1720 °C and 1600 °C was better than 2 °C over
101 a time span of 100 min, which was sufficient for the heat treatment of the tested
102 thermocouples. Due to the high temperatures and the purpose of the heat treatment
103 (the generation of drift within the thermocouples), high levels of thermal stability
104 are not necessary. The same furnaces' thermal stability measured at 960 °C over
105 100 min was better than 0.1 °C. The temperature profile at 960 °C was measured,
106 and the maximum temperature difference over 15 cm (measured from the middle of
107 furnace) was better than 3 °C (0.5 °C over a distance of 10 cm). For the temperature
108 of 1600 °C, the difference measured was better than 6.5 °C, and for 1720 °C, the
109 difference did not exceed 8 °C. This has proven to be sufficient for our measure-
110 ments as the tested thermocouples were placed in a static position, and the signifi-
111 cant factor affecting the measurements was the temperature stability of the furnace.
112 A separate furnace for the measurements at fixed point of Ag was used. This fur-
113 naces thermal characteristic (thermal stability and uniformity) was optimized for the
114 realization of fixed point of Ag accordingly with respect to the ITS-90 [7] and its
115 relevant supplements.

116 For the purpose of the initial thermocouple homogeneity measurements, a stirred
117 vertical oil bath was used. The temperature to which the tested thermocouples
118 were introduced to was 120 °C. The bath's temperature stability at this temperature
119 was better than 0.01 °C with a vertical overall temperature uniformity better than
120 0.02 °C.

121 To determine the long- and short-term drift, two separate measurements were
122 done. These measurements lasted from 8 h (for the short-time exposure to higher
123 temperature of 1720 °C) to 4 months (for the long-time exposure to lower tempera-
124 ture of 1600 °C), and the uncertainty of this difference is very close to the uncer-
125 tainty of one measurement. The whole experiment setup for the long-term and short-
126 term testing is represented by a three-zone horizontal furnace, tested thermocouples,
127 Ag fixed-point cell, calibrated reference Pt/Pd thermocouple and a data recording
128 system.

129 2.2 UME Facilities

130 UME has tested two Type R and two Type S thermocouples. These thermocouples
131 were commercially made with an outer and inner insulation made of high-purity alu-
132 mina (99.7 %), and the total length of the tested thermocouples outer insulation was
133 700 mm for Type R and 800 mm for Type S thermocouples. The outer diameters
134 of sensors were 6 mm for the Type R and 7 mm for Types S with a wall thickness of
135 1 mm. The reference junction was housed in a metal-sheathed capsule.

136 Tests were performed in a commercial horizontal furnace with sufficient homo-
137 geneity and thermal stability. The horizontal profile of this furnace has been meas-
138 ured over a length of 5 cm; the temperature homogeneity was better than 2.5 °C.
139 To further improve the temperature uniformity of the furnace, a cylindrical block
140 made of isolation brick was inserted into the furnace and the thermocouples were
141 placed inside the wells of this block. This has improved the temperature homogene-
142 ity over the same length to 1.9 °C. Silver fixed point was used to check the thermo-
143 electric stability of the tested thermocouples after each test period. The usage of a
144 fixed point instead of a reference thermocouple will enable us to see even the small-
145 est differences in the emf (electro motive force) with benefit of avoiding the aging
146 influence on the reference. The emf of the thermocouples was measured by a cali-
147 brated multimeter device. The whole experiment setup for the long-term and short-
148 term testing is represented by a three-zone horizontal furnace, tested thermocouples,
149 Ag fixed-point cell, calibrated reference Pt/Pd thermocouple and a data recording
150 system.

151 For the purpose of the initial thermocouple homogeneity measurements, a dedi-
152 cated stirred vertical oil bath was used. The temperature to which the tested thermo-
153 couples were introduced to was 100 °C. The bath's temperature stability at this tem-
154 perature was better than 0.01 °C with vertical overall temperature uniformity better
155 than 0.015 °C.

156 2.3 Experimental Procedures

157 The presented study is intended to see the short- and long-time effects of tempera-
158 ture exposure on new noble metal thermocouple of Types B, R and S. The thermo-
159 couples Type R and Type S were annealed for use above the temperature of 800 °C
160 by a 2-h exposure to an isothermal temperature of 1100 °C.

161 The bases for protocol testing are described in [9]. Two different tests were
162 considered:

- 163 • Procedure for testing short-term drift
- 164 • Procedure for testing long-term drift

165 It is expected that these tests will be performed at different temperatures.

166 *Drift: short-term*

167 This test should be applied to a calibrated and properly annealed thermocouple.
168 For the Types R and S, thermocouples are intended to be used above 800 °C, and
169 these should be exposed to a stable and homogenous temperature field for at least
170 2 h and passively cooled at room temperature [10–12]. This is also the case for Type
171 B thermocouples although currently there is no representative information regarding
172 the effectiveness of this procedure. Recommended procedure for determining ther-
173 mocouple short-term drift/thermoelectric stability is as follows:

- 174 1. Calibration of the tested thermocouple at the maximum temperature of intended
175 use and a homogeneity check. (The homogeneity check should be performed by
176 introducing a steep moving temperature gradient along the measuring junction
177 of the thermocouple. This can be realized by emerging the thermocouple from
178 an isothermal environment with a temperature and homogeneity that will not
179 introduce any alternations to the thermoelements.)
- 180 2. Testing the thermocouples at the desired temperature.
- 181 3. The duration of the test is 8 h maximum
- 182 4. Calibration of the thermocouple at the maximum temperature of intended use.
- 183 5. Repeat steps 3–4 three times.

184 The calibration drift is determined as the voltage change (or its temperature
185 equivalent) between the first calibration and the final calibration.

186 *Drift: long-term*

187 This test should be applied to a calibrated and properly annealed thermocouple.
188 Recommended procedure for determining thermocouple long-term drift/thermoelec-
189 tric stability is as follows:

- 190 1. Calibration of the tested thermocouple at the maximum temperature of intended
191 use and a homogeneity check.
- 192 2. Testing of the thermocouples at the desired temperature.
- 193 3. The duration of the test is 4 months at minimum.
- 194 4. Calibration of the tested thermocouple at the maximum temperature of intended
195 use every week during the first month and every 2 weeks for the following
196 months.
- 197 5. The test(s) are finished if
 - 198 (a) The agreed final time is reached, or
 - 199 (b) There is no output from the thermocouple, or

200 (c) The deviation of the tested thermocouple from standard ENIEC 60584-1
201 exceeds 3 times the desired tolerance.

202 The calibration drift is determined as the voltage change (or its temperature
203 equivalent) from the first measurement to the termination.

204 2.3.1 Drift Measurements CMI

205 The drift measurements were performed with four Type B thermocouples. The tested
206 thermocouples homogeneity was initially measured in an oil bath with a set tem-
207 perature of 120 °C from which it was slowly withdrawn from the maximum emer-
208 sion. The length of the thermocouple that was measured in this manner was 300 mm
209 from the tip of the thermocouple (excluding the first 70-mm segment from the tip
210 due to large heat dissipation with low emersion). The purpose of these homogeneity
211 measurements was to detect possible severe defects without the risk of altering the
212 thermocouple homogeneity by high-temperature exposure before the testing cycles.
213 The measured values were only indicative in nature and were not used to form the
214 final uncertainty budget. After these initial measurements, the thermocouples were
215 calibrated in a silver fixed-point cell. After this step, one thermocouple was selected
216 for a short-term drift test. The initially calibrated thermocouple was exposed to a
217 temperature of 1720 °C for a time period of 8 h. After this temperature exposure,
218 a repeated calibration was performed to see the influence of the high-temperature
219 exposure and the possible level of EMF (electromotive force) drift. The calibration
220 was done in the same furnace by comparison with a Pt–Pd reference thermocou-
221 ple in a furnace set to 960 °C. The calibration temperature was set to minimize the
222 introduction of an additional drift to the tested and reference thermocouple. This
223 procedure was repeated three times. The other three thermocouples were investi-
224 gated for long-term drift at 1600 °C for 4 months. The calibrations at fixed point of
225 silver were performed as well. Second run of measurements was performed with one
226 piece of Type R and one piece of Type S thermocouples. The same procedure for
227 short-term drift testing was applied. The temperature used for these tests was set for
228 1600 °C.

229 2.3.2 Drift Measurements UME

230 A number of four noble metal thermocouples (2× R-type and 2× S-type) have been
231 measured for long-term drift (at 1000 °C) and short-term drift (at 1600 °C) tests.
232 The thermocouples dimensions were 880 mm in length with an outer diameter of
233 7 mm with a wall thickness of 1 mm. The thermocouple protective sheath was made
234 out of alumina material.

235 The tested thermocouple homogeneity was initially measured in an oil bath with
236 a set temperature of 100 °C from which it was slowly withdrawn from the maxi-
237 mum emersion. The length of the thermocouple that was measured in this way was
238 320 mm from the tip of the thermocouple (excluding the first 80-mm segment from
239 the tip due to large heat dissipation with low emersion).

240 For long-term drift testing, these thermocouples were first checked at Ag fixed
 241 point and then exposed to a temperature of 1000 °C in a commercial 3-zone Carbo-
 242 lite furnace. During this temperature exposure, the thermocouple voltage was not
 243 measured. The thermocouples were kept at 1000 °C for 1 week and then removed
 244 and checked at Ag fixed point. This process was repeated eleven times. For short-
 245 term drift testing, the thermocouples were kept at 1600 °C for 8 h. Since there was
 246 significant bending in the 30-cm front region of the thermocouples after exposing
 247 to 1600 °C, testing at Ag fixed-point was not possible after they were removed.
 248 So a comparison with a Pt–Pd reference thermocouples in a furnace set to 960 °C
 249 was performed to keep track of any drift. This procedure was repeated three times.
 250 The voltage output from the tested thermocouples was measured by a calibrated
 251 multimeter.

252 3 Results

253 3.1 Drift Testing CMI

254 The calibration results of Type B thermocouples at fixed point of silver were the
 255 following: TC-B I (4497.8 μV), TC-B II (4494.1 μV), TC-B III (4498.0 μV) and
 256 TC-B07/II (4494.0 μV). The first set of short-term test measurements performed on
 257 B I thermocouple is shown in Fig. 1. The two black lines in the figure indicate the
 258 class 2 tolerance limits according to the European standard for thermocouples [8].
 259 The dotted line indicates linear approximation of the drift. The drift rate observed in
 260 our measurements was of -0.27 °C per day at 1720 °C. The several-day exposure
 261 to temperature around 1720 °C has caused a deformation of the ceramic sheath of
 262 the tested thermocouple. This mechanical issue did not influence the behavior of

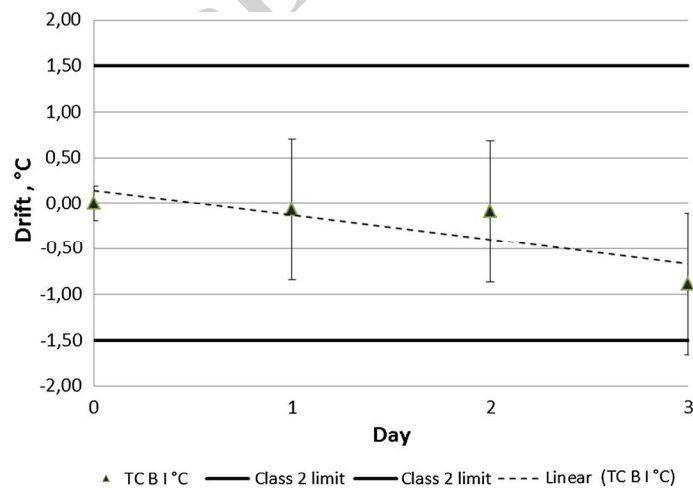


Fig. 1 Short-term drift of Type B thermocouple (exposed to 1720 °C). Drift data obtained by repeated comparison calibration against a Pt/Pd thermocouple at 960 °C

Author Proof

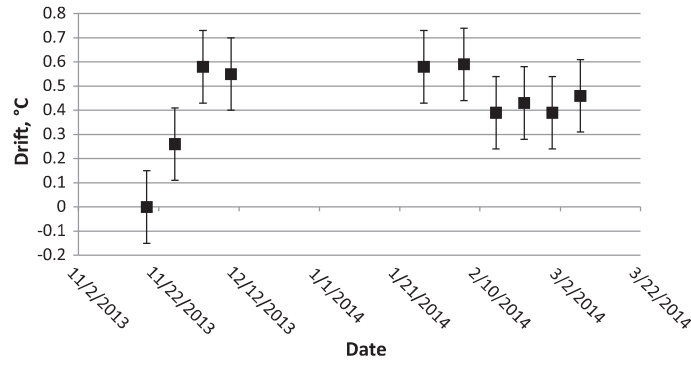


Fig. 2 Long-term drift of Type B II thermocouple (exposed to 1600 °C). Drift data obtained by repeated calibration at fixed point of silver

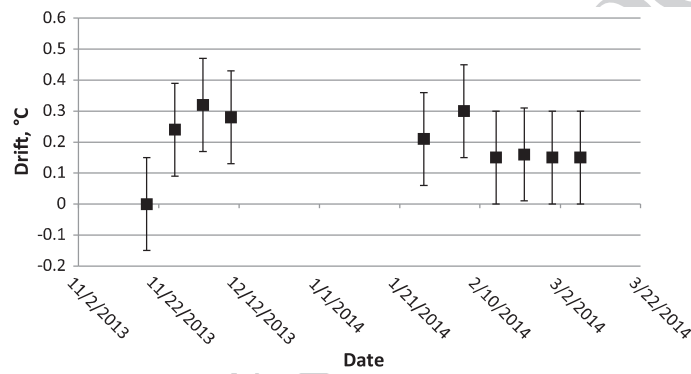


Fig. 3 Long-term drift of Type B III thermocouple (exposed to 1600 °C). Drift data obtained by repeated calibration at fixed point of silver

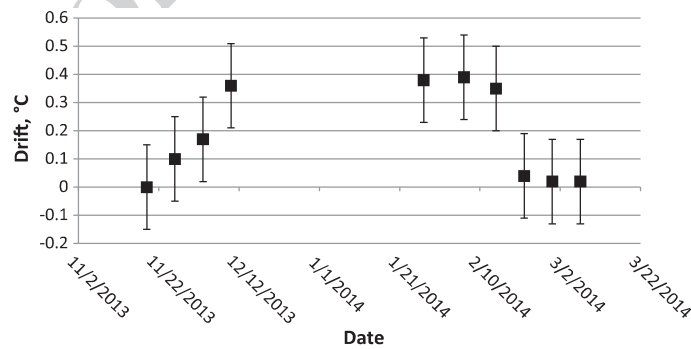


Fig. 4 Long-term drift of Type B 07/II thermocouple (exposed to 1600 °C). Drift data obtained by repeated calibration at fixed point of silver

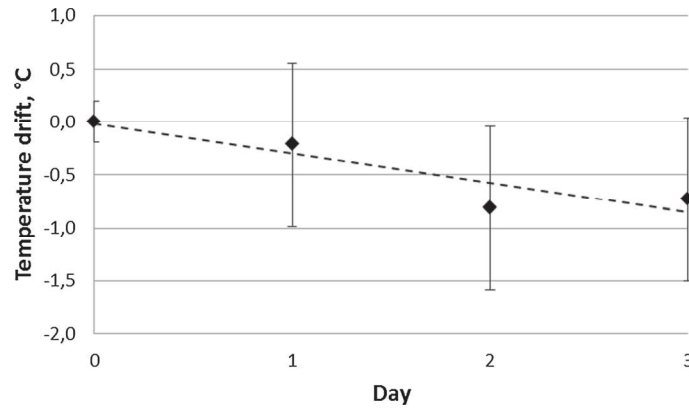


Fig. 5 Short-term drift of Type R thermocouple (exposed to 1600 °C). Drift data obtained by repeated comparison calibration against a Pt/Pd thermocouple at 960 °C

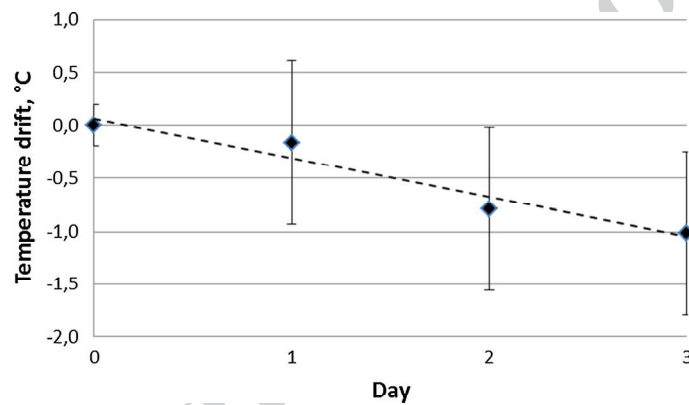


Fig. 6 Short-term drift of Type S thermocouple (exposed to 1600 °C). Drift data obtained by repeated comparison calibration against a Pt/Pd thermocouple at 960 °C

263 the sensor. After these measurements, the long-term investigation was performed on
 264 three Type B thermocouples. The results are shown in Figs. 2, 3, 4. It is necessary
 265 to mention that the class 2 tolerance limit of ± 1.5 °C was not reached after this test.

266 The first calibration of Type R and S thermocouples at fixed point of silver has
 267 given the following results: TC R (9985.3 μ V) and TC S (9143.3 μ V). The short-
 268 term drift test procedure was applied on these Type R and S thermocouples at
 269 1600 °C. At every 8 h, a calibration by comparison at 960 °C was performed with
 270 a calibrated Pt–Pd thermocouple as a standard. The results of these short-term drift
 271 investigation measurements for Type R and S thermocouples are presented in Figs. 5
 272 and 6.

273 The dotted lines indicate linear approximation of the drift at the value of
 274 -0.28 °C (Type R thermocouple) and -0.37 °C (Type S thermocouple) per day at

Table 1 Type R thermocouple (1) thermoelectric stability at Ag fixed point after testing

| Weeks | Average emf (mV) | Average t (°C) |
|----------------|------------------|------------------|
| 0 ^a | 9.9730 | 959.440 |
| 1 ^a | 9.9897 | 960.719 |
| 2 ^a | 9.9814 | 960.084 |
| 3 | 10.0037 | 961.801 |
| 4 | 10.0032 | 961.760 |
| 5 | 10.0025 | 961.711 |
| 6 | 10.0040 | 961.822 |
| 8 | 10.0037 | 961.798 |
| 10 | 10.0019 | 961.666 |

^aMeasurement influenced by the nanovoltmeter thermoelectric stability

Table 2 Type R thermocouple (2) thermoelectric stability at Ag fixed point after testing

| Weeks | Average emf (mV) | Average t (°C) |
|----------------|------------------|------------------|
| 0 ^a | 10.0046 | 961.861 |
| 1 ^a | 10.0039 | 961.814 |
| 2 ^a | 10.0137 | 962.565 |
| 3 | 10.0025 | 961.711 |
| 4 | 10.0031 | 961.756 |
| 5 | 10.0032 | 961.763 |
| 6 | 10.0055 | 961.935 |
| 8 | 10.0044 | 961.851 |
| 10 | 10.0033 | 961.771 |

^aMeasurement influenced by the nanovoltmeter thermoelectric stability

275 1600 °C, respectively. During these short-term drift measurements, a deformation of
 276 the ceramic sheath of the thermocouples occurred due to temperature exposure. This
 277 mechanical issue did not influence the behavior of the sensors.

278 3.2 Drift Testing UME

279 The data presented in this section were obtained by using the procedures described
 280 in Sect. 2.3. The total length of the long-term testing was reduced to 10 week. For
 281 long-term drift test, the thermoelectric stability of Type S and R thermocouples
 282 before and after the temperature exposure is presented in Tables 1 and 2. The first
 283 three measurements at Ag cell presented in these tables were not considered due
 284 to a thermoelectric stability problem of the measurement instrument which can
 285 also be seen from the provided data. This faulty nanovoltmeter was replaced with
 286 another calibrated multimeter, and much stable results were obtained. The first three
 287 measurements are marked and not included in Figs. 7, 8, 9, 10. Each measurement

288 presented within the previously mentioned figures represents the measured value at
 289 the solidification temperatures of Ag fixed point. In order to calculate uncertainty of
 290 measurement, typical standard deviation of the plateau readings (during 20 min or
 291 more) and the standard deviation of melt and freeze average values were quadrati-
 292 cally summed and expanded by a factor of 2 ($k = 2$). Uncertainty values are given
 293 as y-axis error bars. The graphical interpretation of these results can be found in
 294 Figs. 7, 8, 9, 10.

295 For short-term drift test, the thermoelectric stability of Type S and R thermo-
 296 couples was measured as the difference between the tested thermocouples tempera-
 297 ture indication and reference Pt–Pd thermocouple temperature indication at 960 °C
 298 before and after a testing process. The results of these measurements are shown

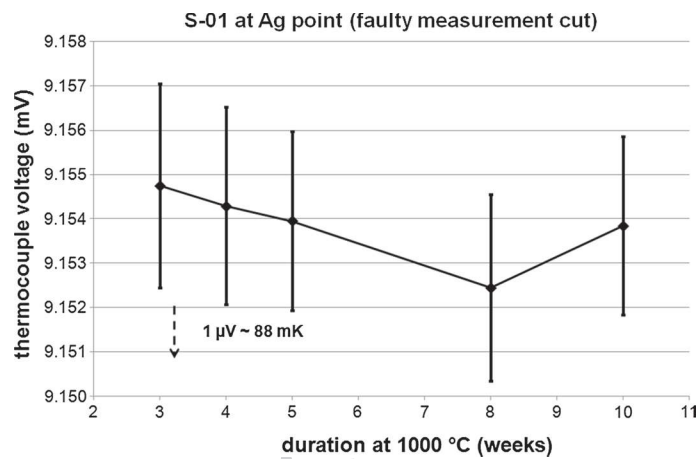


Fig. 7 Type S thermocouple thermoelectric stability (1) at Ag fixed point after testing process

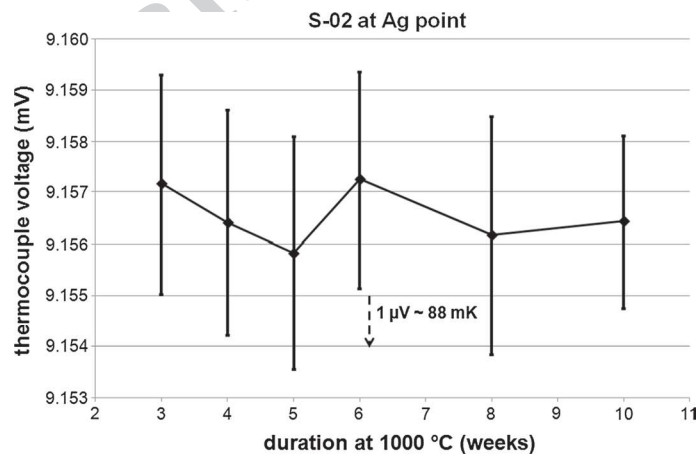


Fig. 8 Type S thermocouple thermoelectric stability (2) at Ag fixed point after testing process

Author Proof

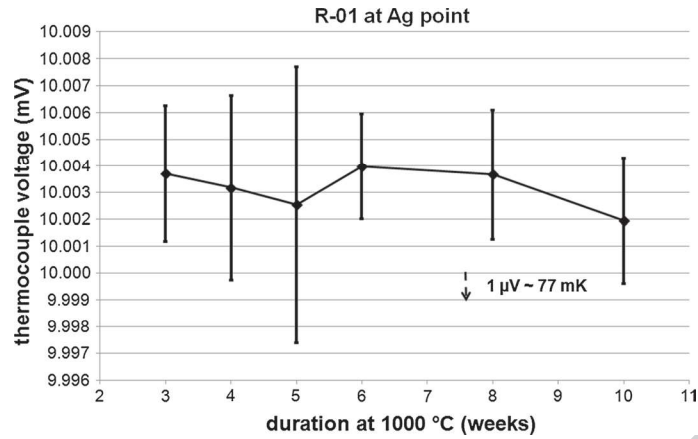


Fig. 9 Type R thermocouple thermoelectric stability (1) at Ag fixed point after testing process

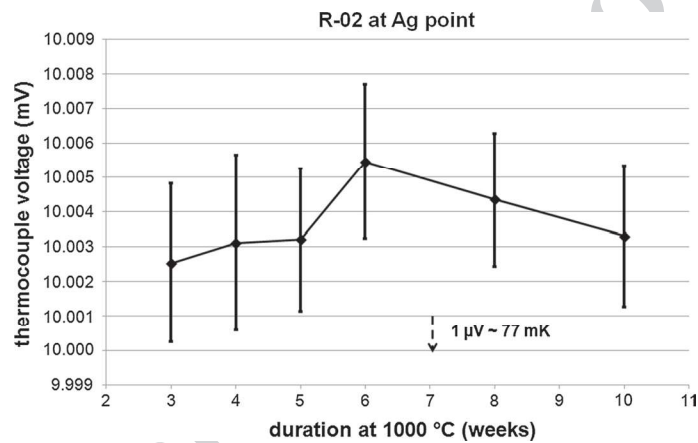


Fig. 10 Type R thermocouple thermoelectric stability (2) at Ag fixed point after testing process

299 in Fig. 11. Here, the zeroth measurement is the initial measurement performed at
 300 960 °C (without any exposure to 1600 °C). The following three measurements were
 301 performed at 960 °C, after 8 h of exposures to 1600 °C.

302 During the testing of the thermocouples at 1600 °C, it was observed that the test
 303 thermocouples were bent at 30-cm front region. This corresponds with the length
 304 of ceramic block which was used to establish better temperature uniformity in the
 305 furnace. If a larger block was utilized over a larger area, this bending would not

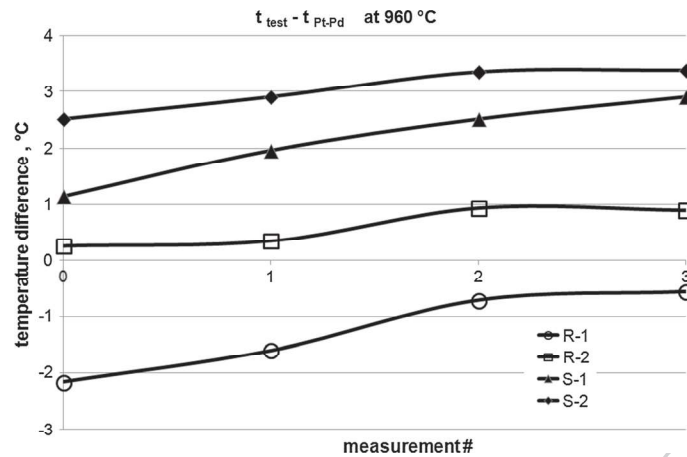


Fig. 11 Thermoelectric stability of R- and S-type thermocouples at 960 °C (as difference from the reference Pt–Pd thermocouple)

306 possibly occur or occur at a larger distance from the tip of thermocouple, and this
 307 would make it possible to have measurements at Ag fixed point. Despite the bend-
 308 ing, the thermoelectric stability of R- and S-type thermocouples after exposing to
 309 1600 °C is quite good (within 2 °C both at 960 °C and at 1600 °C).

310 4 Measurement Uncertainty

311 Within this section, a detailed uncertainty budget of each individual measuring tech-
 312 niques used for the determination of tested thermocouple emf drift by the national
 313 metrological institutes CMI and UME is presented. The uncertainty of the silver
 314 fixed-point measurement performed by CMI can be seen in Table 3, and the uncer-
 315 tainty budget for calibration by comparison at 960 °C is shown in Table 4. The two
 316 calibration methods were used for the long-term tests performed at 1720 °C and
 317 short-term tests performed at 1600 °C, respectively. The overall uncertainty of the
 318 measurement performed by UME, consisting of two different calibration methods
 319 for long- and short-term measurements, can be seen in Table 5. AQ2

320 5 Conclusion

321 This paper shows the results of drift and thermoelectric stability measurements
 322 obtained by the different NMI for Type S, B and R thermocouples. The study was
 323 performed on multiple thermocouples which were exposed to temperatures from
 324 1000 °C to 1720 °C. Their thermoelectric stability was checked using fixed-point

Table 3 Uncertainty budget of silver fixed-point measurements

| Source | z_{\max} | Distribution | κ | u_x | k | u_y | u_y |
|---------------------------------------|-------------------------|--------------|----------|--------------------------|----------------------------------------------|---------------------|--------------------------|
| Realization of fixed point | 1 μV | Normal | 2.000 | 0.500 μV | 1 $\mu\text{V}\cdot\mu\text{V}^{-1}$ | 0.500 μV | 0.044 $^{\circ}\text{C}$ |
| Temperature profile | 0.05 $^{\circ}\text{C}$ | Rectangular | 1.732 | 0.029 $^{\circ}\text{C}$ | 11.4 $\mu\text{V}\cdot^{\circ}\text{C}^{-1}$ | 0.329 μV | 0.029 $^{\circ}\text{C}$ |
| Heat conduction | 0.05 $^{\circ}\text{C}$ | Rectangular | 1.732 | 0.029 $^{\circ}\text{C}$ | 11.4 $\mu\text{V}\cdot^{\circ}\text{C}^{-1}$ | 0.329 μV | 0.029 $^{\circ}\text{C}$ |
| Multimeter measurement | 0.3 μV | Rectangular | 1.732 | 0.173 μV | 1 $\mu\text{V}\cdot\mu\text{V}^{-1}$ | 0.173 μV | 0.015 $^{\circ}\text{C}$ |
| Parasitic thermovoltage. incl. switch | 0.2 μV | Rectangular | 1.732 | 0.115 μV | 1 $\mu\text{V}\cdot\mu\text{V}^{-1}$ | 0.115 μV | 0.010 $^{\circ}\text{C}$ |
| Cold junction | 0.05 $^{\circ}\text{C}$ | Rectangular | 1.732 | 0.029 $^{\circ}\text{C}$ | 5.4 $\mu\text{V}\cdot^{\circ}\text{C}^{-1}$ | 0.156 μV | 0.029 $^{\circ}\text{C}$ |
| Thermocouple homogeneity | 0.1 $^{\circ}\text{C}$ | Rectangular | 1.732 | 0.058 $^{\circ}\text{C}$ | 11.4 $\mu\text{V}\cdot^{\circ}\text{C}^{-1}$ | 0.658 μV | 0.058 $^{\circ}\text{C}$ |
| Thermocouple hysteresis | 0.1 $^{\circ}\text{C}$ | Rectangular | 1.732 | 0.058 $^{\circ}\text{C}$ | 11.4 $\mu\text{V}\cdot^{\circ}\text{C}^{-1}$ | 0.658 μV | 0.058 $^{\circ}\text{C}$ |
| Type A | 0.05 $^{\circ}\text{C}$ | Rectangular | 1.732 | 0.029 $^{\circ}\text{C}$ | 11.4 $\mu\text{V}\cdot^{\circ}\text{C}^{-1}$ | 0.329 μV | 0.029 $^{\circ}\text{C}$ |
| Combined uncertainty | | | | | | 1.695 μV | 0.152 $^{\circ}\text{C}$ |
| Coverage factor | | | | | | 2 | 2 |
| Expanded uncertainty | | | | | | 3.40 μV | 0.30 $^{\circ}\text{C}$ |

z_{\max} —uncertainty component value, κ —value pertaining to a given probability distribution, u_x —standard uncertainty for respective distribution, k —sensitivity coefficient, u_y —respective standard uncertainty converted into μV and $^{\circ}\text{C}$

Author Proof

325 cells after a certain aging period was reached. It was clearly shows that the method-
 326 ology used is suitable for this type of exploration and can be done not only at level
 327 of NMI. The results are not indication of any thermocouple capabilities of Types R,
 328 S and B, but can be used as one example of the thermocouple behavior. Long-term
 329 drift and short-term drift tests have been performed on R-, S- and B-type thermo-
 330 couples by different metrology institutes. The main results are that the long-term
 331 drift of Type B thermocouples at 1600 $^{\circ}\text{C}$ and R-, S-type thermocouples at 1000 $^{\circ}\text{C}$
 332 are much smaller than thermoelectric stability declared by EN 60584-1 standard tol-
 333 erance classes after 10-week and 4-month exposure. The short-term thermoelectric
 334 stability of R- and S-type thermocouples when exposed to 1600 $^{\circ}\text{C}$ for a repetition
 335 of 8-h periods has been within 2 $^{\circ}\text{C}$ when measured by comparison with a reference
 336 Pt–Pd thermocouple at 960 $^{\circ}\text{C}$. The short-term thermoelectric stability of R- and
 337 S-type thermocouples when exposed to 1600 $^{\circ}\text{C}$ for a repetition of 8-h periods has
 338 been within 1 $^{\circ}\text{C}$ when measured by comparison with a reference Pt–Pd thermocou-
 339 ple at 960 $^{\circ}\text{C}$. The short-term thermoelectric stability of B-type thermocouple when
 340 exposed to 1720 $^{\circ}\text{C}$ for repetition of 8 h has been within 1 $^{\circ}\text{C}$.

341 This works goal was to define a procedure for an investigation of drift behavior of
 342 noble metal thermocouples at high temperature.

343

Table 4 Uncertainty budget of a calibration by comparison at 960 °C

| Source | z_{\max} | Distribution | κ | u_x | k | u_y | u_y |
|-------------------------------------------|-------------------|--------------|----------|---------------------|--------------------------------------|---------------------|----------|
| Calibration temperature | 0.3 °C | Normal | 2.000 | 0.150 °C | 11.4 $\mu\text{V}\cdot\text{C}^{-1}$ | 1.710 μV | 0.150 °C |
| Temperature profile and thermal stability | 0.6 °C | Rectangular | 1.732 | 0.346 °C | 11.4 $\mu\text{V}\cdot\text{C}^{-1}$ | 3.949 μV | 0.346 °C |
| Heat conduction | 0.05 °C | Rectangular | 1.732 | 0.029 °C | 11.4 $\mu\text{V}\cdot\text{C}^{-1}$ | 0.329 μV | 0.029 °C |
| Multimeter measurement | 0.3 μV | Rectangular | 1.732 | 0.173 μV | 1 $\mu\text{V}\cdot\mu\text{V}^{-1}$ | 0.173 μV | 0.015 °C |
| Parasitic thermovoltage, incl. switch | 0.2 μV | Rectangular | 1.732 | 0.115 μV | 1 $\mu\text{V}\cdot\mu\text{V}^{-1}$ | 0.115 μV | 0.010 °C |
| Cold junction | 0.05 °C | Rectangular | 1.732 | 0.029 °C | 5.4 $\mu\text{V}\cdot\text{C}^{-1}$ | 0.156 μV | 0.029 °C |
| Thermocouple homogeneity | 0.1 °C | Rectangular | 1.732 | 0.058 °C | 11.4 $\mu\text{V}\cdot\text{C}^{-1}$ | 0.658 μV | 0.058 °C |
| Thermocouple hysteresis | 0.1 °C | Rectangular | 1.732 | 0.058 °C | 11.4 $\mu\text{V}\cdot\text{C}^{-1}$ | 0.658 μV | 0.058 °C |
| Type A | 0.05 °C | Rectangular | 1.732 | 0.029 °C | 11.4 $\mu\text{V}\cdot\text{C}^{-1}$ | 0.329 μV | 0.029 °C |
| Combined uncertainty | | | | | | 5.044 μV | 0.444 °C |
| Coverage factor | | | | | | 2 | 2 |
| Expanded uncertainty | | | | | | 10.09 μV | 0.88 °C |

z_{\max} —uncertainty component value, κ —value pertaining to a given probability distribution, u_x —standard uncertainty for respective distribution, k —sensitivity coefficient, u_y —respective standard uncertainty converted into μV and °C

Author Proof

Table 5 Overall uncertainty for long- and short-term measurements performed by UME

| Measurement type | Uncertainty component | Value (μV) | Value (mK) |
|-------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------|-------------------------|-------------------|
| Typical uncertainty budget for long-term drift tests (measurements performed at Ag fixed point) | Typical thermoelectric stability of a single plateau (maximum of melt and drift) | 2.08 | 160 |
| | Typical repeatability of plateaus (standard deviation of melt and freeze) | 2.27 | 175 |
| | Cell uniformity | 2.60 | 200 |
| | Multimeter calibration | 0.25 | 19 |
| | Total uncertainty ($k = 1$) | 4.04 ^a | 311 ^a |
| | Expanded uncertainty ($k = 2$) | 8.08 ^a | 622 ^a |
| Uncertainty budget for short-term drift tests (measurements performed at furnace at 960 °C) | Typical thermoelectric stability during a single measurement | 2.72 | 200 |
| | Furnace uniformity | 8.17 | 600 |
| | Multimeter calibration | 0.25 | 19 |
| | Total uncertainty ($k = 1$) | 8.62 ^a | 633 ^a |
| | Expanded uncertainty ($k = 2$) | 17.23 ^a | 1266 ^a |

^aThe presented uncertainty values represent a typical uncertainty for the performed measurements. For each individual measurement and sensor, this value may vary due to the changing budget components like thermoelectric stability which depend on individual sensors. The specific values of uncertainty are presented within Figs. 7, 8, 9.

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