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INRIM–NMC Comparison of Pt/Pd Calibration Above the Ag Point

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Abstract A comparison of a Pt/Pd calibration above the Ag point between the INRIM and NMC was arranged with the aims of evaluating measurement systems and exploiting the potential of the Pt/Pd thermocouples. Two commercial Pt/Pd thermocouples were used as transfer thermometers. A calibration method using a blackbody cavity as a transfer source and a radiation thermometer as a reference thermometer was adopted in both institutes. The T_{90} carried by the radiation thermometers is established by an extrapolation technique for INRIM and by scale realization according to ITS-90 definition for NMC and, therefore, this exercise is also a useful comparison of different approaches to disseminate T_{90} above the Ag point. The comparison results are presented and analyzed.

Keywords Blackbody cavities · Fixed points · Pt/Pd thermocouple · Radiation thermometer

1 Introduction

A Pt/Pd thermocouple is the most common contact thermometer for accurate temperature measurements above 1000 °C. Its calibration above the temperature of the silver point (962 °C), directly traceable to the ITS-90, requires a radiation thermometer as a reference. INRIM developed a transfer temperature source consisting of a SiC blackbody cavity and a thermometer well heated by a multi-zone furnace able to

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assure good axial temperature distribution. As such an arrangement allows calibration of Pt/Pd thermocouples against a radiation thermometer in an accurate manner [1], NMC also developed a similar facility to calibrate high-temperature thermocouples. With the aims of validating the respective measuring systems and fully exploiting the potential of Pt/Pd thermocouples up to 1500 °C, INRIM and NMC arranged a comparison of a Pt/Pd thermocouple and radiation thermometer based on such facilities.

Four Pt/Pd thermocouples are arranged for the comparison. Two of them are fabricated at INRIM, and the other two are commercially available thermocouples belonging to NMC. The measurement range of the two INRIM thermocouples is up to 1500 °C, while that of the NMC ones is up to 1300 °C due to the temperature limits recommended by their manufacturer. However, due to some unforeseen delay, the two INRIM thermocouples are still in the process of measurement and it is not possible to present the results in this article. Therefore, only the results of the two NMC thermocouples are discussed.

The two NMC thermocouples were first characterized at NMC by performing inhomogeneity measurements and Ag-point calibrations. Calibration against the NMC reference radiation thermometer from 960 °C to 1300 °C was then followed. After the thermocouples were calibrated at the Ag point and against the radiation thermometer at INRIM, the same measurements were repeated at NMC.

At INRIM, a radiation thermometer carrying a multi-fixed-point calibration was used to define T_{90} of the transfer source cavity [2]. The thermometer was the primary standard of INRIM, based on a Hamamatsu S2592-03 silicon photodiode operating in an unbiased mode, with a built-in Peltier element allowing the temperature of the detector to be controlled at 10 °C. The instrument was used in a spectral configuration with an interference filter centered at 950 nm with a bandpass of 70 nm, which allows the instrument to be operated from the Zn point up. The thermometer was calibrated at the Zn, Al, Ag, and Cu fixed points, and an interpolating equation approximating Planck's equation was used to relate the signals to the temperatures. In the present exercise, the temperatures were defined by either an interpolation or extrapolation approach, depending on whether they were lower or higher with respect to T_{Cu}, respectively.

At NMC, a radiation thermometer with a working wavelength of 0.65 μm calibrated according to the ITS-90 definition was used instead. Consequently, the exercise was also a useful comparison of different approaches to disseminate T_{90} above the silver point.

2 Measurements

2.1 Pt/Pd Thermocouples and Their Inhomogeneities

The two NMC thermocouples, identified as CS094-01 and CS094-02, have prefixed re-crystallized alumina protection tubes with an outer diameter of 7 mm and a length of 600 mm for the hot junction and stainless tubes for the reference junction. Platinum and palladium wires, purities of 99.999 % and 99.99 %, respectively, of diameter 0.5 mm and length 1800 mm are used as thermocouple elements. According to the manufacturer, the thermocouples were constructed using the techniques developed by

NMIJ [3]. Before construction, the thermocouple wires were electrically annealed at 1200 °C for 10 h. The thermocouples were furnace annealed at 1100 °C for 3 h and at 1030 °C for 100 h after the thermocouples were constructed. Both thermocouples were newly purchased before the comparison started.

Upon arrival of the two thermocouples at NMC, their inhomogeneity was scanned in a liquid bath against a standard platinum resistance thermometer at about 200 °C. The measurements were repeated after a first calibration up to 1300 °C at NMC (before sending to INRIM), upon arrival from INRIM, and when all measurements were completed.

2.2 Calibration of Two NMC Thermocouples at Ag Point

The Ag-point calibration at NMC was used as a stability check in addition to the inhomogeneity check. The calibration was done before and after each calibration up to 1300 °C. The Ag fixed point used is a commercially available mini-fixed-point cell with a fixed-point cell immersion of about 140 mm and overall furnace immersion of about 290 mm [4]. It has been validated by a Ag fixed-point cell used for high-temperature SPRT calibration using an Au/Pt thermocouple as a transfer thermometer and is used routinely for thermocouple calibrations. The immersion characteristic of the thermocouples in the Ag fixed-point cell was tested by insertion and withdrawal step-by-step during plateaus.

The two thermocouples were calibrated at the Ag point at INRIM before calibration up to 1300 °C using the INRIM fixed-point cell normally used for calibrating high-temperature SPRTs [5]. The design of the cell assures an immersion of about 180 mm in the liquid metal.

2.3 Calibration of Thermocouples Against Radiation Thermometer Using Blackbody Cavity as Transfer Source

Details of the INRIM thermocouple calibration facility against a radiation thermometer using a blackbody cavity as a transfer source are described in [1]. The facility is an improvement of what was used for developing the reference function of a Pt/Pd thermocouple from 800 °C to 1500 °C [6]. The furnace consists of three independent heating zones with heating elements made of molybdenum disilicite ($MoSi_2$) for operation up to 1800 °C. A specially designed home-made blackbody cavity is used as a transfer source for measurements with the radiation thermometer. As shown in Fig. 1, the blackbody cavity has a cylindrical shape, 100 mm long with a diameter of 10 mm and an aperture diaphragm of 4 mm. It consists of a composite material, obtained by bonding silicon carbide and alumina powders. In order to minimize the error due to a possible axial temperature gradient, the thermocouple under measurement was positioned just behind the back wall of the blackbody cavity, as closely as possible. The blackbody back wall is positioned exactly in the middle of the furnace, 45 cm from the edge.

A Pt/Pd thermocouple was inserted into the lateral thermocouple well at a radial distance of 3 cm from the center, and temperature profiles were measured from 960 °C

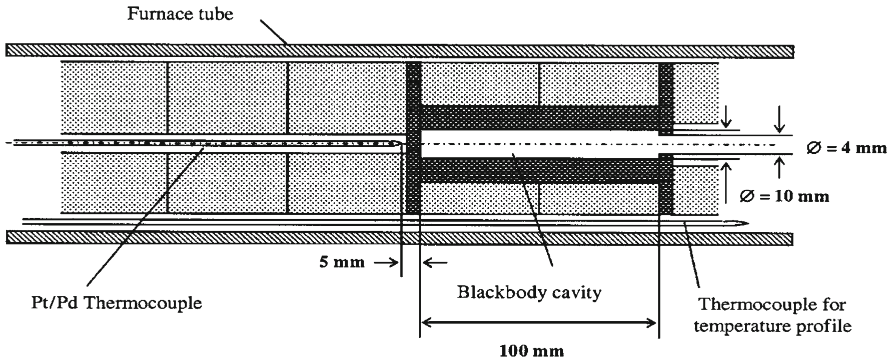

Fig. 1 Geometrical arrangement of the blackbody insert for measurements with radiation thermometer for INRIM setup

to 1300 °C. It was found that, for a length of 100 mm across the middle of the furnace, i.e., ±50 mm with respect to the back wall of the cavity, the temperatures are controlled within ±0.25 °C.

Even though the furnace was characterized in terms of the axial temperature distribution and proper setting parameters were available, a check was made prior to each measurement by extracting the thermocouple from its measurement position. The signals proved to be stable, for the first 3 cm, within less than 0.1 °C at all temperatures. Two rounds of measurements were carried out for each of the NMC thermocouples.

The NMC blackbody cavity and thermometer well have a very similar design as that of INRIM. They were manufactured by INRIM. The furnace used is also similar, but has better temperature control. Some minor differences are: (a) the cavity is positioned slightly toward the back of the furnace which results in a thermocouple immersion of about 40 cm instead of 45 cm in the INRIM setup and (b) no aperture diaphragm is used. Prior to the comparison, the furnace uniformity was assessed by two calibrated type B thermocouples, one inserted into the thermometer well (B1)

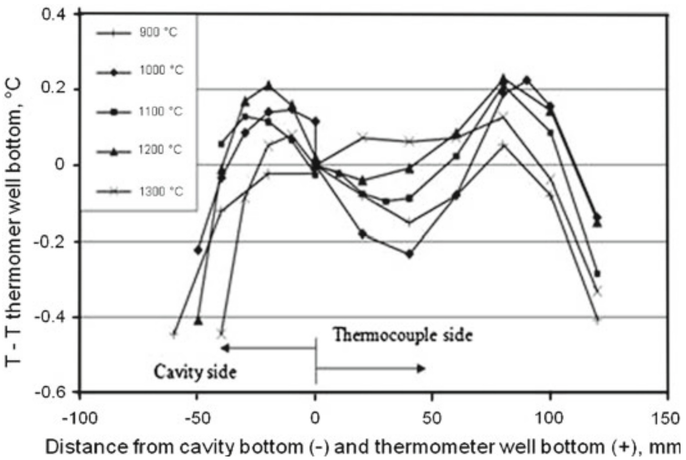

Fig. 2 Temperature uniformity along the cavity and thermometer well for NMC setup

and the other inserted into the cavity (B2). The results are shown graphically in Fig. 2 where the temperature difference, with respect to that of the thermometer well bottom, is drawn according to the distance to the cavity bottom, for B2 (indicated as a negative value) and to the thermometer well bottom for B1 (indicated as a positive value). As can be seen, the temperature uniformity is within ±0.2 °C within about 100 mm in the thermometer well from 900 °C to 1300 °C.

As stated earlier, calibration of the two thermocouples started at NMC. To minimize any possible deterioration, limited measurements (one round for each thermocouple) from 1000 °C to 1300 °C were performed before sending them to INRIM. The total burning time was about 10 h for each of the thermocouples. On return, more extensive measurements were carried out. These measurements included one round for CS-9401 and two rounds for CS-9402 from 960 °C to 1300 °C, and immersion checks which were carried out at the end of the measurements at each tested temperature using mostly thermocouple CS-9402. The obtained results are in line with those of the uniformity tests prior to the comparison. The accumulated burning time above 1000 °C was about 15 h for CS-9401 and 35 h for CS-9402. Including the burning time at INRIM, the overall burning time was 43 h for CS-9401 and 56 h for CS-9402.

3 Results

3.1 Results of Inhomogeneity Tests

The results of inhomogeneity scans are shown in Fig. 3a and b for CS094-01 and CS094-02, respectively. As demonstrated in the figures, CS094-01 was very stable and no significant deterioration was found in the whole process of the comparison exercise. On the other hand, for CS094-02, deteriorations were observed especially at the end of the comparison. This thermocouple was stable before sending to INRIM; and upon returning from INRIM, no significant change in terms of inhomogeneity along the thermocouple wire was found, although a slight shift in terms of the emf difference with respect to the Pt/Pd reference function [7] was observed. However, the thermocouple was significantly deteriorated after all measurements, including two runs of calibration against the radiation thermometer and the Ag-point measurements after each run of calibration, were completed. This deterioration explains the further drift at the Ag point found after the second run of calibration as discussed in the next section. A 30 min anneal at 1070 °C was attempted, but it caused further deterioration rather than reversal.

3.2 NMC Measurement Results at the Ag Point

Thermocouple CS094-01 demonstrated very good homogeneity and plateau reproducibility in the whole process of the comparison. The standard deviation of a total of seven plateau values at a fixed immersion depth, measured before and after each heat-up to 1300 °C, is 0.1 μV which is equivalent to about 5 mK, or a peak-to-peak variation of 0.28 μV (15 mK). As an example, one withdrawal and one insertion immersion curves measured during a single plateau are plotted in Fig. 4. The peak-to-peak varia-

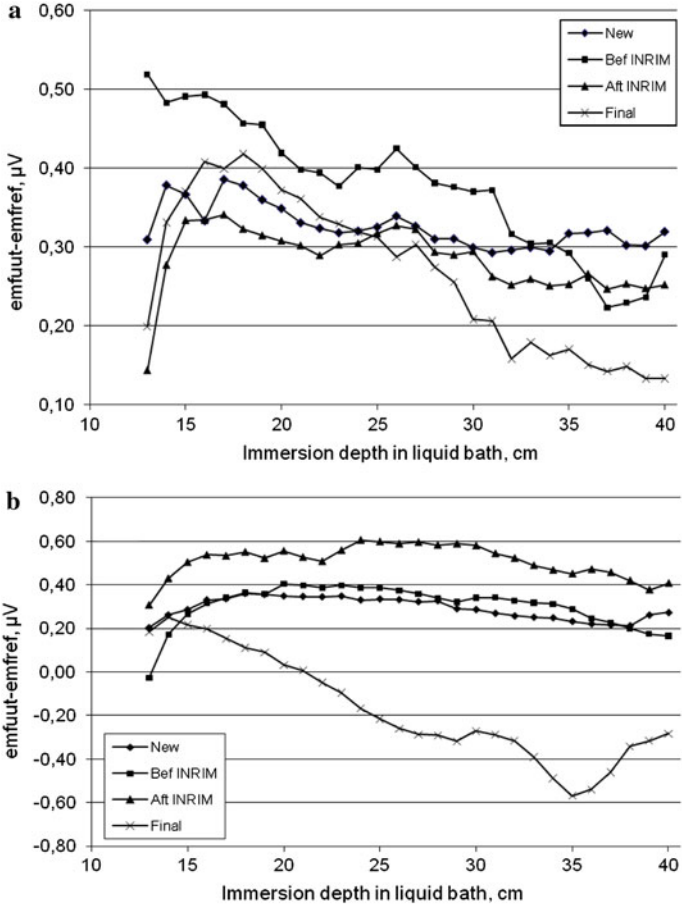

Fig. 3 Inhomogeneity scans of the thermocouples used in the comparison (emfuut) with respect to the Pt/Pd reference function (emfref): (a) for CS094-01 and (b) for CS094-02

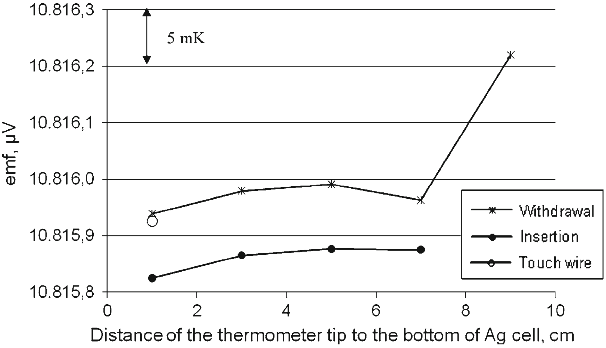

Fig. 4 Immersion profile of thermocouple CS094-01 in the Ag fixed-point cell during a plateau realized at NMC

tion from immersion of 1 cm to 7 cm from the Ag fixed-point cell bottom, an indication of inhomogeneity, is about 0.05 μV. The difference between the withdrawal and the insertion, an indication of hysteresis, is about 0.1 μV. However, it is worthwhile to note that this difference may not be indicative of some sort of instability of the thermocouple. We noticed during the plateau realization that if the thermocouple wire, which leads the thermocouple handle to the cold junction, was touched, the thermocouple reading would not return to its original value even when the measurement system was re-stabilized after the disturbance caused by the touch. For example, during the immersion test, we started with withdrawal and followed by insertion. After the collection of data at 1 cm away from the cell bottom during the insertion process, we did not do anything but touch the wire. The thermocouple value jumped to the withdrawal value at the same immersion depth immediately after the touch, which is indicated in Fig. 4. This disturbance was observed for both thermocouples at NMC (not tested at INRIM). Some studies were carried out, and the manufacturer was also consulted. However, it is still not clear about the cause of this effect but it is quite small and is therefore considered as part of the overall reproducibility of the thermocouple. As can be seen in Fig. 4, the thermocouple reading increases significantly at about 9 cm away from the fixed-point cell bottom. This is due to the influence of the furnace temperature as the mini-fixed-point cell is quite short.

The immersion characteristics of CS094-02 are very similar to that of CS094-01 during the initial tests before sending to INRIM. Upon returning from INRIM, the Ag-point value had drifted lower by about 1.8 μV (0.09 °C) as compared with the previous result and this drift was stable until after the second run of calibration up to 1300 °C. A further drift of about 1.4 μV (0.07 °C) in the same direction was observed after the second run, and at the same time, the thermocouple showed a very strange behavior of taking an excessively long time to stabilize during the Ag-point plateau. The inhomogeneity scan performed later revealed that the thermocouple had deteriorated significantly.

Measurement results of both thermocouples, except that of CS094-02 after the second run calibration up to 1300 °C, are given in Table 1, in terms of the emf difference with respect to the Pt/Pd reference function. All NMC and INRIM measurement results are graphically summarized in Fig. 5 where the drift of CS094-02 is clearly shown.

3.3 Measurement Results for Radiation Thermometer Calibration

3.3.1 NMC Calibration Result

Measurement results before sending to and after returning from INRIM are tabulated in Table 1. The result of one run, or average of two runs where applicable, is graphically shown for each thermocouple in Fig. 6. As can be seen easily from Fig. 6, the emf differences with respect to the Pt/Pd reference function are significantly different between before and after measurements. While the measurement results at 960 °C after INRIM agree well with the Ag fixed-point calibration with a difference of about 0.11 °C for both thermocouples, the measurement results at 960 °C before INRIM deviate about 0.4 °C from the Ag fixed-point calibration. This indicates that the before

Table 1 Calibration results at Ag point and by radiation thermometer for both INRIM and NMC presented in terms of differences with respect to the reference function of Pt/Pd thermocouple at the respective test temperature

T (°C)	Difference to ref. NMC before (μV)		Difference to ref. INRIM (μV)		Difference to ref. NMC after (μV)		Difference between NMC after and INRIM (μV)
Ag point:	2.53		2.93		2.53		−0.40
Radiation:	Round 1	Round 2	Round 1	Round 2	Round 1		
CS094-01							
960	8.68	8.37	2.89	1.73	0.34		−1.97
1000		10.63			1.66		
1100		11.55	2.32	0.53	2.78		1.35
1200		13.91	5.22	3.77	5.91		1.42
1300		19.43	2.55	1.22	7.74		5.85
Ag point:	2.61		−1.14		0.84		1.98
Radiation:	Round 1		Round 1	Round 2	Round 1	Round 2	
CS094-02							
960			1.31	0.88	−0.78	−1.78	−2.38
1000	10.57				−0.85		
1100	10.98		0.13	−0.33	1.43	0.38	1.00
1200	14.38		4.08	2.47	3.54	2.57	−0.22
1300	18.93		2.31	0.40	6.23	5.64	4.58

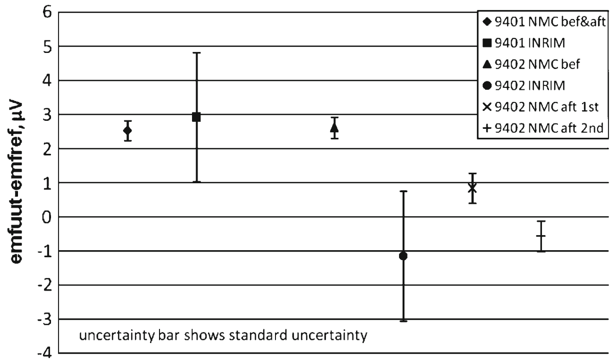

Fig. 5 Results of calibration against Ag point presented in terms of emf differences referenced to the Pt/Pd reference function for two laboratories

INRIM calibration is erroneous. As a matter of fact, when the deviation between the Ag-point calibration and radiation thermometer calibration was observed during the measurement, investigations with other thermocouples were carried out after the two thermocouples were sent to INRIM due to time constraints and concern of prolonged burning time on the two transfer standards. Nothing abnormal was found except the

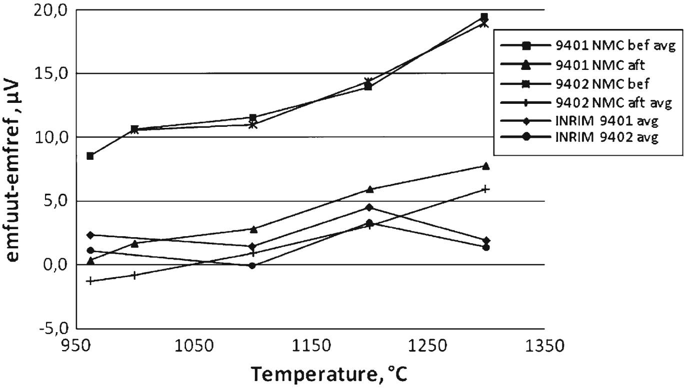

Fig. 6 Results of calibration against radiation thermometer presented in terms of emf differences referenced to the Pt/Pd reference function for two laboratories

stabilization time for the multi-zone furnace. This furnace is very stable when it is stabilized (a few hundredths of a degree Celsius at all test temperatures as indicated by the thermocouples). However, it takes a very long time to obtain this condition. Due to such, at some stage, the temperature changes very slowly and appears stable as seen by the thermocouple or radiation thermometer; however, the system is not stabilized yet due to different thermal lags of the cavity and the thermometer well. Therefore, it was very likely that insufficient time was allowed during the before measurements while trying to reduce the burning time of the thermocouples.

As discussed earlier in Sect. 3.2, the Ag-point calibration value of CS094-02 after returning from INRIM is lowered by about 1.8 µV as compared to that before sending it to INRIM. This drift is consistent with the radiation thermometer calibration at 960 °C and at higher temperatures. This conclusion can be drawn by looking at the differences between the two thermocouples. In the before measurement, there are almost no differences between the two thermocouples at all tested temperatures, as in the Ag-point calibration. On the other hand, there is an almost constant difference between the two thermocouples in the after measurement with the CS094-02 lower by about 2.1 µV on average, which agrees well with the 1.8 µV drift at the Ag-point calibration.

Finally, the two rounds of measurement of thermocouple CS094-02 after returning from INRIM repeated very well with an agreement of 0.05 °C or better for all tested temperatures despite the deterioration found during the final inhomogeneity scan.

3.3.2 INRIM Calibration Result

The INRIM calibration results are given in Table 1 as well. Similarly, an average of two runs for each thermocouple is shown graphically in Fig. 6. As indicated in Table 1, the measurements repeated very nicely in the two rounds of measurements for both thermocouples with a maximum difference of about 0.08 °C. Excellent agreement between the Ag-point calibration and radiation thermometer calibration at 960 °C was obtained for thermocouple CS094-01, which was only 0.03 °C, and a slightly larger, but yet good agreement of 0.12 °C was obtained for thermocouple CS094-02.

3.4 Measurement Uncertainty

The estimated uncertainty for the INRIM measurements by comparison with the radiation thermometer directly refer to the uncertainty budget discussed in [1]. As the magnitude of the various uncertainty components u_i, namely, the repeatability, radiation thermometer calibration, inhomogeneity, SSE, emissivity, positioning, emf measurements, etc. varies for each data point, values of the combined standard uncertainty span the temperature range, and they vary linearly, between 0.13 °C at 960 °C and 0.25 °C at 1300 °C. As for the uncertainty at the Ag point at INRIM, a standard uncertainty of 0.1 °C was estimated. Such a large figure originates from taking into account possible effects due to inhomogeneity which was not directly measured at INRIM.

The standard measurement uncertainties on the Ag-point measurement at NMC were estimated to be 0.29 μV, 0.31 μV, and 0.44 μV for thermocouple CS094-01 and thermocouple CS094-02 before INRIM and after INRIM, respectively. An average variation from immersion of 1 cm to 7 cm from the Ag fixed-point cell bottom of all measured plateaus is considered as an uncertainty that originated from the inhomogeneity. The slightly larger uncertainty of thermocouple CS094-02 after INRIM is due to a larger immersion error found during the plateau measurement. The uncertainty components include: (a) fixed-point temperature uncertainty; (b) uncertainty that originated from the thermocouple reading, such as nano-voltmeter calibration uncertainty, repeatability of measurement, any possible contact effect due to the use of scanner and reference junction uncertainty, etc.; (c) reproducibility of several plateaus; and (d) inhomogeneity/immersion error of the thermocouple (based on the scans in the Ag cell).

The result of the uncertainty estimation for the radiation thermometer calibration is given in Table 2. The uncertainty components are: (a) uncertainty that originated from the radiation thermometer reading including ITS-90 realization uncertainty,

Table 2 NMC measurement uncertainty of calibration against radiation thermometer of the two thermocouples

Uncertainty components	Estimated standard uncertainty at various temperatures				
	960 °C	1000 °C	1100 °C	1200 °C	1300 °C
Radiation thermometer reading (°C)	0.11	0.11	0.14	0.17	0.23
Thermocouple reading (μV)	0.65	0.65	0.56	0.50	0.66
Furnace uniformity (°C)	0.02	0.02	0.07	0.05	0.05
Inhomogeneity of thermocouple (μV)	1.06	1.13	1.34	1.55	1.77
Reproducibility of measurement (°C)	0.05	0.05	0.05	0.05	0.05
Combined standard uncertainty (μV)	2.6	2.8	3.8	4.5	6.0
Combined standard uncertainty (°C)	0.14	0.14	0.18	0.21	0.26

repeatability of measurement and uncertainty that originated from the positioning of the radiation thermometer; (b) uncertainty that originated from the thermocouple reading (see (b) of the Ag-point uncertainty for the sources of the uncertainty); (c) uncertainty due to the furnace uniformity during measurement; (d) inhomogeneity of the thermocouple based on scans in the bath and in the furnace (worst case); and (e) reproducibility of the two rounds of measurements.

3.5 Comparison of the INRIM and NMC Results

The agreement between the INRIM and the NMC measurement results is generally good for both Ag-point measurements and calibration by a radiation thermometer.

For Ag-point agreement, in the case of thermocouple CS094-01, the difference from the two laboratories is only 0.4 µV in emf, equivalent to 21 mK, as evidenced by Table 1 and Fig. 5. In the case of thermocouple CS094-02, comparison of the results is not straightforward because of the drifting of the thermocouple. As discussed earlier in Sect. 3.3.1, the two thermocouples demonstrated very similar emf values at all tested temperatures before they were sent to INRIM. However, after returning from INRIM, the emf of thermocouple CS094-02 deviated from thermocouple CS094-01 constantly by about 2.1 µV lower. A similar deviation was observed for the INRIM measurements, i.e., the emf value of CS094-02 was constantly lower than that of CS094-01 by an average of about 1.1 µV. This could possibly lead to a conclusion that the thermocouple had already changed since it arrived at INRIM. If this assumption is taken, we can then compare the NMC Ag measurement results immediately after returning from INRIM with the INRIM one. In this case, the difference is about 2.0 µV, equivalent to 0.10 °C.

As discussed in Sect. 3.3.1, the NMC calibration results by a radiation thermometer before the thermocouples were sent to INRIM were erroneous due to insufficient stabilization time allowed to the furnace. Therefore, only the measurement results after the thermocouple returned from INRIM were considered for the comparison. In this case, as can be inferred from the results in Table 1 and the uncertainty values reported for INRIM and those in Table 2 for NMC, the agreement is within the standard measurement uncertainty of both laboratories at all tested temperatures for both thermocouples. Despite such good agreement, it is not difficult to notice that the shapes of the emf curves are different between INRIM and NMC as shown in Fig. 6. There is a trend in the curves for both NMC thermocouples, while the INRIM ones are almost flat. Several efforts have been devoted in finding possible reasons, including re-measuring the effective wavelength of the radiation thermometer used. However, no cause has been found to date.

3.6 Conclusions and Future Activities

In conclusion, good agreement in the comparison of Pt/Pd thermocouples above the Ag point between INRIM and NMC using the two NMC transfer thermocouples is obtained. The comparison results show that the measurement setups of both institutes are suitable for accurate calibration of high-temperature thermocouples against radiation thermometers. This conclusion is, in particular, important for NMC as its system has been only recently set up. It also confirms indirectly the good agreement of the dissemination of T_{90} above the Ag temperature in the two institutes despite different approaches of either ITS-90 realization or approximation used. During the comparison, one of the thermocouples has been extremely stable and can be used to compare the Ag-point realization at the two institutes in an accurate manner even after burning up to 1300 °C. This confirms that the Pt/Pd thermocouple can be very stable and suitable for comparison and accurate studies up to at least 1300 °C. Future studies with the two INRIM thermocouples originally planned for this study as mentioned in Sect. 1 will verify this suitability up to 1500 °C. On the other hand, another thermometer used in the comparison and handled in the same manner showed a drift in its emf and deteriorated significantly in its homogeneity. It is not clear if its initial drift in emf is caused by the transportation or for some other reason, but it can be deduced that further deterioration is caused by repeated burning up to 1300 °C. Further study will be carried out to investigate any other possible causes of drift and deterioration. As mentioned in Sect. 3.5, despite the good agreement obtained, there is a slight tendency that the thermocouple's emf difference with respect to the Pt/Pd reference function increases according to the test temperature. This might be an undiscovered systematic error. This doubt will be possibly cleared by the future studies using the two INRIM thermocouples up to 1500 °C.

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