Final report of comparison "APMP.T-S16"

APMP Regional Comparison of Type R (Pt-Pt13%Rh) Thermocouples calibration at Cu, Co-C and Pd fixed Points

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1 INTRODUCTION

Platinum (Pt) based thermocouples are widely used to provide traceability to accredited laboratories at high temperatures (>1100 °C) and are routinely calibrated up to 1560 °C by many NMIs within APMP (Asia-Pacific Metrology Program) . Although a successful comparison to 1100 ^{o}C has been performed (APMP.T-S1-04 [1, 2]), no equivalent comparison has so far been made over the higher temperature range.

In general, a thermocouple calibration consists of measuring the thermocouple emf, E, at several values of tip temperature and comparing the emf with the corresponding value, E_{ref}, given by a defined reference function. Different NMIs have adopted different procedures to calibrate thermocouples in the temperature range (1100 °C to 1560 °C): such as gold (Au, 1064.18 °C) and palladium (Pd 1554.82 °C) [3] 'melt wire' techniques (in both air and argon), mini fixed point of copper (Cu) and cobalt-carbon eutectic (Co-C) and palladium-carbon eutectic (Pd-C) points, and comparison with reference thermocouples and/or radiation thermometers.

A key function of this comparison is to examine the equivalence of the diverse techniques used by the APMP NMIs for the calibration of client's Pt-based thermocouples over 1100-1560 ^{o}C.

The pilot lab (NMIA) surveyed the facilities used by the APMP laboratories in this high temperature range for the calibration of Pt-based thermocouples to help guide the design of the comparison. Nine potential participants responded: 7 laboratories use Au and Pd-melt wire techniques and Co-C eutectic fixed points; 8 use a Cu fixed point; four labs have facilities of using the Pd-C eutectic point. As the previous comparison, APMP.T-S1-04, was conducted from 0 °C to 1100 °C, this comparison started from Cu-point (1084.62 °C), but it was optional. Based on work done by Jahan et al, as the inhomogeneity of a thermocouple drastically changed when used in Pd-C crucible [4], so it was decided that the Pd-C fixed point would not be used in this comparison.

2 SUMMARY OF COMPARISON PROCESS

An inverted star comparison was used to achieve a quicker comparison and to reduce the work for the pilot lab (NMIA). The pilot lab constructed 8 Type R thermocouples and measured the inhomogeneity of each thermocouple. They were then transported to the participating laboratories in July - August 2017. All thermocouples arrived safely to the participating laboratories, without any breakage. Each lab calibrated that particular thermocouple using their own procedures and then sent it back to the pilot lab. All thermocouples arrived unbroken at the pilot lab, except one from NIM (2017-05), which was broken after the measurement completed but before sending. The pilot lab (NMIA) replaced the broken insulator carefully with a new prebaked insulator. Two thermocouples insulator were heavily bent due to long time use at high temperatures. Those insulators had to be replaced by the pilot lab before measurements could be carried out (2017-02) and (2017-08).

All 8 thermocouples were measured for inhomogeneity and annealed to 1100 °C and quenched by the pilot lab. Then all 8 thermocouples were calibrated at the Cu fixed point, Co-C fixed point and Pd melting point according to pilot lab's procedure. After calibration, the thermocouples were sent back to the participating labs for the final measurement. All thermocouples were annealed again at 1100 °C and quenched before sending. The measurement sequence of the comparison is described schematically in diagram below (Figure 1).

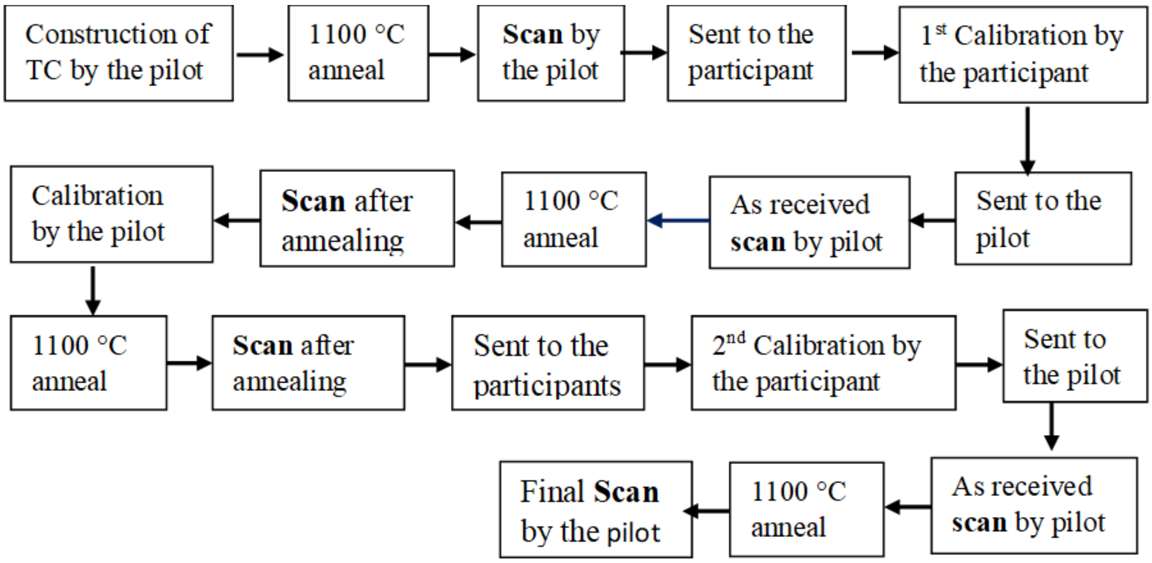


Figure 1. Measurement sequence of the comparison.

All thermocouples arrived safely at the participating laboratories for the second round except NMIJ/AIST and NMISA. The insulator of these two thermocouples (2017-09) & (2017-11) were broken during transport. After communicating with the pilot lab, the insulator was replaced by a new prebaked insulator by the corresponding lab.

The APMP Thermocouple Intercomparison protocol, version 6, which was circulated in (April 2017, and approved in May 2017), is given in **Appendix A**. In the protocol, it was mentioned that the thermocouples are at **1100 °C Quenched state and calibration should be lower to higher temperatures**. Each laboratory calibrated the given thermocouple at Cu, Co-C fixed point and Pd melting point and though some labs used comparison methods. The instruments used in the measurement of the comparison by the participating laboratories are given in **Appendix B**. The raw calibration data from the participating laboratories are given in **Appendix C**. The calibration procedure used by the individual laboratory is given in **Appendix D**. The uncertainty submitted by the participants are given in **Appendix E** (only one set).

3 MEASUREMENT SCHEDULE

There was a measurement schedule set in the protocol, however it was delayed with respect to protocol due to COVID-19. The comparison measurement sequences was as follows:

April-May'2017 – Protocol circulated and approved.
May-June'2017 – Inhomogeneity measurement of all Thermocouples by the pilot.
July-August'2017 – Artefacts were sent to participating laboratories.
November-December'2017 – Measurement done by the pilot lab with a help of a staff from NIM, China. During that time only 5 thermocouples has been measured and 3 thermocouples were not arrived in time.
2018-2020 – During this period, pilot lab completed the rest of the measurements of the comparison and artefacts were send to the participating laboratories, after receiving their initial results, as described in the protocol.

One exception is that NPLI, India has not submitted their initial results until August' 2022 and taken the thermocouple on January 2023. NPLI submitted their final measurement results on 30 March 2023.

4 DESCRIPTION OF THE ARTEFACTS

The artefacts were Type R thermocouples, selected from a batch of thermocouples used before in APMP comparison, **APMP.T-S1-04**. These thermocouples were selected based on inhomogeneity measurement after suitable high temperature annealing. The thermocouples were made previously as follows:
Each thermocouple was constructed from 1400 mm long Pt and Pt13%Rh wires of 0.5 mm diameter, purchased from Sigmund Cohn Corp. (USA). The insulators used were high purity, alumina, purchased from Ceramic Oxide Fabricators (Australia). They were 750 mm long and 4.1 mm in diameter with two bores of 1.1 mm diameter. They were prebaked at 1150 °C for 6 hours.

For NMIJ/AIST, the thermocouple wires were 2270 mm long, upon special request from the lab to make the thermocouple with longer wires but with same insulator length.

The thermocouples wires were bare wire annealed at 1400 °C for 1 hour and 1100°C for 1 hour. After assembled into the insulator, they were given another 1 hour anneal at 1100°C and 16 hours anneal at 450 °C.

5 MEASUREMENT PROCESS BY THE PARTICIPATING LABS

Table 1 gives a summary of the calibration procedure used by each of the participants in the comparison. The details of the procedures used by each laboratory is given in Appendix D.

Table 1. Calibration procedure used by the Participating laboratories.

Name of Laboratory	**Calibration Process**
NMIA – Australia	Cu mini fixed point, Co-C mini fixed point and Pd minicoil technique in argon atmosphere.
NIM – China	Fixed points used : Cu fixed point, Co-C fixed point and Pd melt wire technique in argon atmosphere.
NPLI – India	Fixed points used : Cu fixed point, Co-C fixed point and Pd melt wire technique in argon atmosphere.
BSN – Indonesia	By comparison with type B and Pt/Pd thermocouples and find the values at 1084 °C, 1324 °C and 1554 °C
NMIJ/AIST – Japan	Cu point was not done, Co-C fixed point and Pd melt wire technique in air.
KRISS – Korea	Cu fixed point, Co-C fixed point and Pd-fixed point.
NMC, A*STAR – Singapore	By comparison with a radiation thermometer using a black body furnace and Cu fixed point for final measurement.
NMISA – South Africa	Cu fixed point and Pd melt wire technique in air
NIMT – Thailand	Cu fixed point, Co-C fixed point and Pd melt wire technique in air.

Note: As some labs had not performed fixed point measurements but reported calibration by comparison with a reference standard and provided results at Cu, Co-C and Pd point temperatures, in this document the terms 'Cu point', 'Co-C point' and 'Pd point' are used to represent the values given by the participating labs at those points and same terminology is used for both the direct measured and calculated values in this report.

6 MEASUREMENT OF THE THERMOCOUPLES BY THE PILOT LAB

This is an inverted-star comparison: participating lab performed initial measurements and then the Pilot lab measured those thermocouples. The participating lab then remeasured those thermocouples. To assess the impact of irreversible changes in the thermocouples due to exposure to high temperatures, the inhomogeneity of the thermocouple was measured by the pilot lab at different stages of the comparison. At each stage, before sending the thermocouple to the participating lab, all thermocouples were annealed at 1100 °C for an hour and quenched, and the inhomogeneity of all thermocouples measured at 200 °C by the pilot lab.

After receiving the artefacts from each lab, the measurement by the pilot lab are as follows:

i) the thermocouples were scanned 'As received' at 200 °C in an oil bath [5].
ii) Annealed at 1100 °C for an hour and quenched and scan again.
iii) Measured at Cu-mini fixed point, using NMIA's mini Cu cell [6, 7]
iv) Annealed at 1100 °C for 45 minutes and quenched.
v) Measured at Co-C mini fixed point [8]
vi) Annealed at 1100 °C for 45 minutes and quenched.

vii) Measured at Pd melting point using 'minicoil' technique in argon atmosphere [9].
viii) Annealed again at 1100 °C and quenched.
ix) Sent to the participating lab for the final measurement (noted: the result of the initial measurements had to be submitted before thermocouple was sent for final measurement).

7 MEASUREMENT RESULTS

In this section the measurement results and uncertainties provided by the participants are presented. The values of expanded uncertainty U_{95} provided by the participants and pilot **are without** a term for inhomogeneity. For the pilot laboratory, the uncorrelated uncertainty is also provided to facilitate a reduced uncertainty for lab-lab differences. The uncorrelated uncertainty excludes the two main correlated uncertainty contributions from the temperature assigned to the pilot-lab's fixed-point cell or melt-wire: i.e. the term for assigned ITS-90 cell temperature and the uncertainty estimated from the metal quality, as these two systematic errors will be constant over the period of the comparison. **All** the other terms, related to the method, instrumentation and type-A variance etc are **included** in the uncorrelated uncertainty estimate: please refer to the NMIA uncertainty analysis in Appendix E-1. This is because any systematic temperature error in the pilot lab's temperature scale is fully correlated between all the participants for the calculation of the Lab-Pilot (X_i) value. The full pilot uncertainty (correlated & uncorrelated) is of course used for the later weighting for the reference value. This approach removes potential double-counting of these terms and reduces the uncertainty for all participants.

7.1. Cu point temperature results

In the protocol, measurement of Cu point was optional, as it was covered in previous comparison APMP. T-S1-04. However, all labs measured the thermocouples at the Cu fixed point, except NMIJ/AIST, Japan. Also note that for A*STAR and BSN the values at the Cu temperature were measured using comparison techniques. The values of E-E_{ref} (where E is the measured emf of the TC and $Eref$ is the reference emf) and U_{95} provided by the participating labs are presented in table 2, including the pilot lab's values.

The difference between the initial and final measurements by the participants is well within the laboratory's stated measurement uncertainty, except for NPLI, where the difference is slightly larger than their claimed uncertainty, but still within the combined initial and final uncertainty.

Table 2. E-E_{ref} values at Cu point temperature.

Lab Name	Average, E-E_{ref} / µV		NMIA values E-E_{ref} / µV	U_{95} for NMIA (#uncorrelated terms only)/µV
	Initial (±U_{95})	Final (±U_{95})		
NPLI	0.07 (±3.29)	-4.04 (±3.25)	-0.75	±1.09 (#0.94)
NIM	-1.00 (±2.99)	-0.73 (±2.99)	-2.72	±1.09 (#0.94)
NMC, A*STAR	2.39 (±5.95)*	0.843 (±5.99)*	-2.03	±1.09 (#0.94)
NIMT	-0.60 (±1.12)	-0.80 (±1.12)	-0.80	±1.09 (#0.94)
BSN	-7.80 (±11)*	-6.20 (±17) *	-2.37	±1.09 (#0.94)
NMIJ/AIST	-	-	-1.90	±1.09 (#0.94)
KRISS	-1.36 (±1.18)	-1.50 (±0.43)	-3.94	±1.09 (#0.94)
NMISA	-3.30 (±1.87)	-3.29 (±1.52)	-2.49	±1.09 (#0.94)

* These measurements were done by comparison, whereas other labs used a fixed point.

7.2. Co-C Point temperature results

Five participating laboratories measured their own Co-C fixed point. Two labs, NMC, A*STAR, Singapore and BSN, Indonesia supplied the values of E-E_{ref} at 1324.0 °C, based on calibration by comparison. NMISA, South Africa did not provide values at Co-C fixed point. The values of E-E_{ref} of all thermocouples are given in table 3. The reference temperature of Co-C crucible, as reported by the participant is also included in the table.

The difference between the initial and final measurements by each participants is well within in their stated measurement uncertainty. Although the NPLI difference is slightly over their claimed uncertainty is still within the combined initial & final uncertainty.

Table 3. E-E$_{ref}$ values at Co-C point temperature

Lab Name	Temperature of Co-C point/°C	E-E_{ref} / µV		NMIA values E-E_{ref} / µV	U_{95} for NMIA (#uncorrelated terms only)/µV
		Initial (±U_{95})	Final (±U_{95})		
NPLI	1323.93	1.28 (±5.13)	-5.49 (±5.14)	-6.22	±7.11 (#1.15)
NIM	1324.00	-1.20 (±5.48)	-2.50 (5.48)	-5.82	±7.11 (#1.15)
NMC,A*STAR	1324.00	0.27 (±7.83)	-1.86 (±7.79)	-6.45	±7.11 (#1.15)
NIMT	1324.06	-7.60 (±6.38)	-7.60 (±6.38)	-4.75	±7.11 (#1.15)
BSN	1324.00	-11.0 (±15)	-12.00 (±22)	-5.47	±7.11 (#1.15)
NMIJ/AIST	1324.00	-5.70 (±7.4)	-5.60 (±7.4)	-5.95	±7.11 (#1.15)
KRISS	1324.10	-7.50 (±11.3)	-8.20 (±11.3)	-8.15	±7.11 (#1.15)
NMISA	1324.10	-	-	-6.44	±7.11 (#1.15)

7.3. Pd point temperature results

The calibration of thermocouples at the Pd melting point has been done by all participants. However laboratories had adopted several different techniques to determine E-E_{ref} at Pd melting point, as described in section 5. Two labs, NIM – China, NPLI – India, used Pd melt wire technique in argon atmosphere, considering reference melting point of Pd is 1554.8 °C. Three labs, NMIJ/AIST – Japan, NIMT – Thailand and NMISA – South Africa, has measured Pd melting point in air environment and used reference temperature at Pd melting point of 1553.5 °C. One lab KRISS – South Korea, used Pd fixed point cell, of measured temperature of 1559.1°C. Two other labs, NMC, A*STAR - Singapore and BSN – Indonesia measured Pd point by comparison method with reference standard of radiation thermometer and Type B thermocouple respectively. BSN - Indonesia only supplied an initial measurement value at Pd point.

The pilot lab NMIA – Australia used Pd melting point in argon atmosphere (1554.82 °C) as its ITS-90 reference point [3]. The values of E-E_{ref} and U_{95} values are given in table 4, together with the reference temperature used by each laboratory. The change between the initial and final measurements by the participant is well within their stated measurement uncertainty.

Table 4. $E\text{-}E_{ref}$ values and uncertainties at Pd point temperature

Lab	Reference Temperature	$E\text{-}E_{ref}$ / µV		NMIA $E\text{-}E_{ref}$/ µV	U_{95} for NMIA (#uncorrelated terms only)/µV
		Initial (±U_{95})	Final (±U_{95})		
NPLI	1554.8	-1.78 (±6.07)	-5.93 (±6.09)	-6.80	±2.37 (±1.54)
NIM	1554.8	-5.10 (±8.21)	-8.00 (±8.21)	-5.90	±2.37 (±1.54)
NMC,A*STAR	1554.0	1.47 (±8.5)	1.99 (8.5)	-7.10	±2.37 (±1.54)
NIMT	1553.5	-4.40 (±11.74)	-6.30 (±11.74)	-4.30	±2.37 (±1.54)
BSN	1554.0	-28.00 (±32)	n/a	-4.30	±2.37 (±1.54)
NMIJ/AIST	1553.5	-12.30 (±6.5)	-11.90 (±6.5)	-6.00	±2.37 (±1.54)
KRISS	1559.1	-12.10 (±16.8)	-8.60 (±16.8)	-8.40	±2.37 (±1.54)
NMISA	1553.5	-9.09 (±12.68)	-5.62 (±12.3)	-6.20	±2.37 (±1.54)

8 THERMOCOUPLE LENGTH CHANGES DURING THE CALIBRATION.

Laboratories using the melt wire technique need to cut off the melt-wire-affected tip for each measurement, shortening the thermocouple wires. This shortening of the wires brings wire with potentially less heat treatment into the temperature gradient zone of the furnaces (in which the thermocouple EMF is generated). This is expected to have a negligible effect if the change in length is small compared to the furnace zone lengths.

To assess this, the length of the wires was measured at different stages of the comparison, typically within ±5 mm. Table 5 summarizes the length of the thermocouple wires of each thermocouple at different stages of the comparison. We observe that the thermocouple wires were shortened by between 30 mm and 50 mm, except for TC(2017-10), which was only shortened by 10 mm. This change in length is small compared to the length of thermocouple wires considered in the calculation of the uncertainty contribution from thermocouple inhomogeneity.

Table 5: Length of thermocouple as measured by the pilot.

Thermocouple Serial Number	Length of thermocouple / mm			
	before sending to participant	as received from participant	Before sending to participant	as received from participant
2017-02	1385	1365	1348	1330*
2017-05	1395	1376	1357	1340
2017-06	1395	1380	1363	1350*
2017-07	1390	1380	1370	1355
2017-08	1395	1390	1370	1370
2017-09	2270	2252	2238	2233
2017-10	1390	1390	1380	1375
2017-11	1400	1375	1352	1320*

*TC was not sent to pilot, length was measured by the participating lab

9 THERMOCOUPLE INHOMOGENEITY MESUREMENT RESULTS

The thermoelectric homogeneity of each thermocouple was assessed at 200 °C, using NMIA's oil-bath scan facility [5] at various stages of the comparison. The "initial" thermoelectric signature of all the thermocouples in the comparison is given in Figure 2, showing the excellent homogeneity of the thermocouples prior to the comparison.

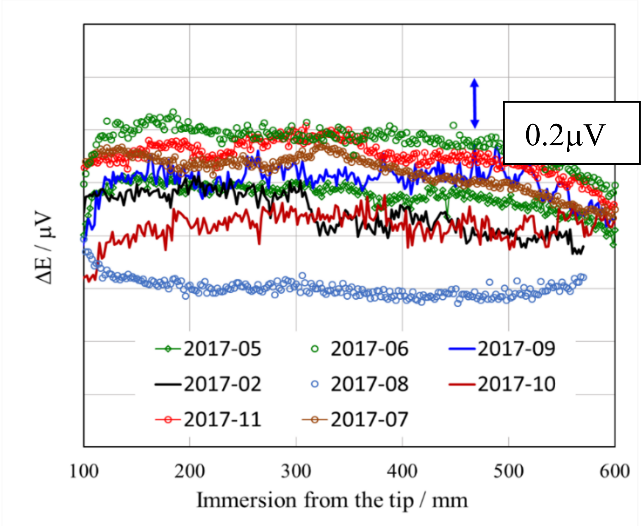


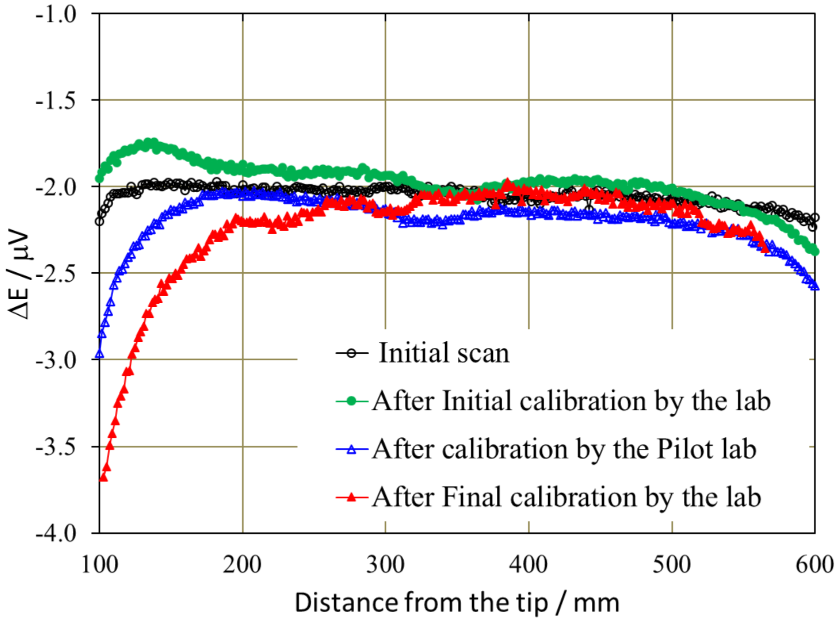
Figure 2. The initial inhomogeneity of all 8 thermocouples. *ΔE* is the change of EMF as a function of position of thermocouples in the temperature gradient zone.

The inhomogeneity of a thermocouple is given by the equation:

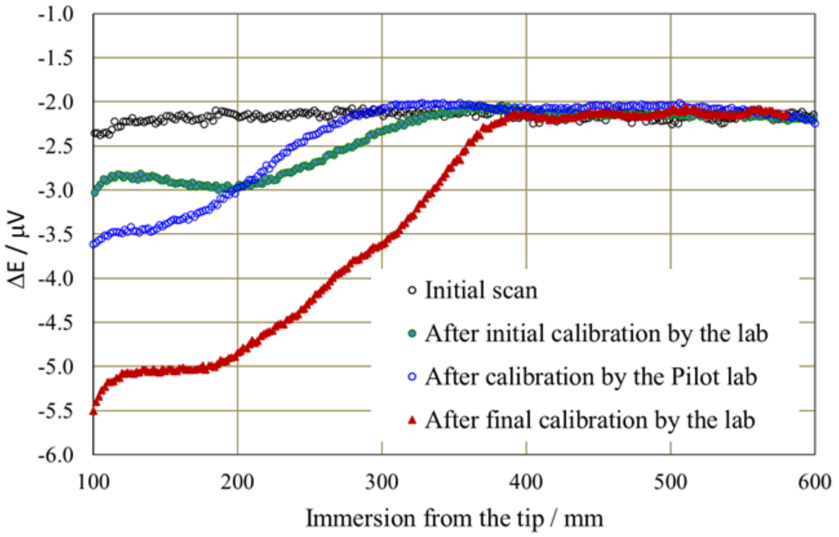
$$\textbf{Inhomogeneity} = [\Delta EMF/(EMF_{Tm} - EMF_{Tamb})]/2*100\% \qquad (1)$$

where ΔEMF is the maximum change of measured *EMF* as a function of immersion length of the TC, and EMF_{Tm} is the EMF at the measured oil bath temperature, T_m=200 °C and EMF_{Tamb} is the *EMF* at ambient temperature $T_{amb}\sim 21°C$. As the current scan facility of NMIA is unable to accurately scan the section of thermocouple between the tip and 100 mm due to conduction errors at these small immersions, we have limited calculation to the region between 100 mm and 500 mm. Table 6 shows the inhomogeneity of each thermocouple, over this region, as calculated using equation 1, from the thermoelectric scans.
The initial "before sending" inhomogeneity shows that all thermocouples have an excellent homogeneity of below ±0.01%, which is typical of new high -quality thermocouple wire, and similar to the values found in APMP.T-S1-04 [1]

The inhomogeneity of all thermocouples was found to have increased significantly after calibration. Figure 3 shows example homogeneity scans of two thermocouples TC 2017-05 (A) and TC 2017-10(B). This is different to the previous comparison of type-R thermocouples up to 1100 °C, APMP.T-S1-04 [1], where change in inhomogeneity (after annealing was applied) was found to be insignificant.



(A)



(B)

Figure 3. Inhomogeneity scan (in annealed state) of two thermocouples after each calibration. (A)TC 2017-05 (B) TC 2017-10

The measured inhomogeneity values of all thermocouples at various stages of comparison are given in Table 6. TCs (2017-10, 2017-08 and 2017-05) showed largest change in inhomogeneity of ≤ ±0.10%, whereas, TCs (2017-07 and 2017-09) showed comparatively small increase of inhomogeneity after calibration. The inhomogeneity of 3 thermocouples (2017-02, 2017-06 and 2017-11) could not be measured at the end of comparison, as the corresponding labs had not sent them back to the pilot for inhomogeneity measurement.

Table 6. Summary of the inhomogeneity values from 100 to 500 mm from the tip.

Thermocouple Serial Number	Inhomogeneity / %					
	before sending to participant	as received from participant	after annealing	before sending to participant	as received from participant	after annealing
2017-02	± 0.009	±0.052	± 0.034	**±0.059**	Not received	-
2017-05	± 0.009	±0.027	± 0.012	**±0.026**	±0.075	±0.052
2017-06	± 0.009	±0.027	± 0.018	**±0.024**	Not received	-
2017-07	± 0.009	±0.026	± 0.009	**±0.011**	±0.037	±0.016
2017-08	± 0.010	±0.022	± 0.020	**±0.034**	±0.073	±0.076
2017-09	± 0.009	±0.018	± 0.014	**±0.020**	±0.033	±0.025
2017-10	± 0.009	±0.041	± 0.033	**±0.055**	±0.110	±0.110
2017-11	± 0.010	±0.052	± 0.019	**±0.055**	*Not received*	-

As the thermocouples were calibrated by each participant in the annealed state, we note that it is the changes in the "inhomogeneity after annealing" that are relvant in the calculation of uncertainties for the comparison here.

The inhomogeneity values in table 6, are provided only to illustrate the typical changes that occur in the thermocouples during the comparison. Simply using these values directly will lead to a significant overestimate of the uncertainty associated with thermocouple inhomogeneity, as they cover the large region of the thermocouple (100 – 500 mm) and it is only the region of the thermocouple where the furnace gradients differ that will be affected by inhomogeneity. In the comparison here, the furnace gradients are very similar, so only a smaller region of the thermocouple need be considered, and the impact of inhomogeneity will be much smaller: whereas for furnaces with very different gradients, the influence will remain large.

In the comparison here, inhomogeneity was assessed at multiple steps during the comparison.
The authors decided to use the value after the calibration by the pilot (before-sending-to-participant highlighted in table 6) as representative of the changes in homogeneity incurred by heat-treatment at both the pilot and participant. (We also note that not all TCs have final inhomogeneity measurements, as they were not returned to the pilot for measurement.)

10 COMPARISON DATA ANALYSIS

10.1. Uncertainty arising from Thermocouple Inhomogeneity

As part of the comparison protocol, participants were required to provide furnace gradient data to the pilot to enable the pilot to assess the impact of thermocouple inhomogeneity on the measured lab-pilot differences. Table 7 shows the gradient zone of the furnaces used by the participating laboratories has been supplied and the values as supplied for A to B and A to C (referred to Fig. 1 in the protocol in Appendix A) are given in Table 7. Figures 5A, 5B and 5C present this data graphically.

Table 7. The immersion length (mm) for 90% and 10% of the temperature-step in as calculated from the data provided by the participants, and the pilot lab.

Lab Name	Cu Point furnace		Co-C Point furnace		Pd Point furnace	
	Tip to 90% of T, A to B	Tip to 10% of T, A to C	Tip to 90% of T, A to B	Tip to 10% of T, A to C	Tip to 90% of T, A to B	Tip to 10% of T, A to C
NPLI	340	470	250	390	320	450
NIM	395	503	286	598	154	357
NMC,A*STAR	268	398	293	398	297	398
NIMT	350	500	400	550	60	200
BSN	280	330	280	330	280	330
NMIJ/AIST	-	-	326	550	195	370
KRISS	370	510	280	510	310	500
NMISA	389.1	525.9	-	-	45.3	174.2
NMIA	306	432	263	482	291	505

There is not much difference in the furnace gradient zones for Cu and Co-C point measurements, so for calculation simplicity, the region showed by dotted blue lines in Figures 5A and 5B is used. However, for Pd point measurement the furnace gradient zones varied widely (Figure 5C). Consequently, the inhomogeneity is calculated individually for the appropriate length of the thermocouple, depending on the gradient zone it experienced in the participating lab and also in the pilot lab.

Table 8. Calculated inhomogeneity values for each thermocouple over the length used in the relevant participant's and pilot's furnaces.

Lab Name	Inhomogeneity %		
	Cu Point	Co-C Point	Pd Point
NPLI	±0.007%	±0.011%	±0.013%
NIM	±0.004%	±0.006%	±0.007%
NMC, A*STAR	±0.004%	±0.006%	±0.003%
NIMT	±0.007%	±0.009%	±0.013%
BSN	±0.006%	±0.006%	±0.006%
NMIJ/AIST	±0.004%	±0.007%	±0.008%
KRISS	±0.013%	±0.017%	±0.004%
NMISA	±0.005%	±0.006%	±0.050%

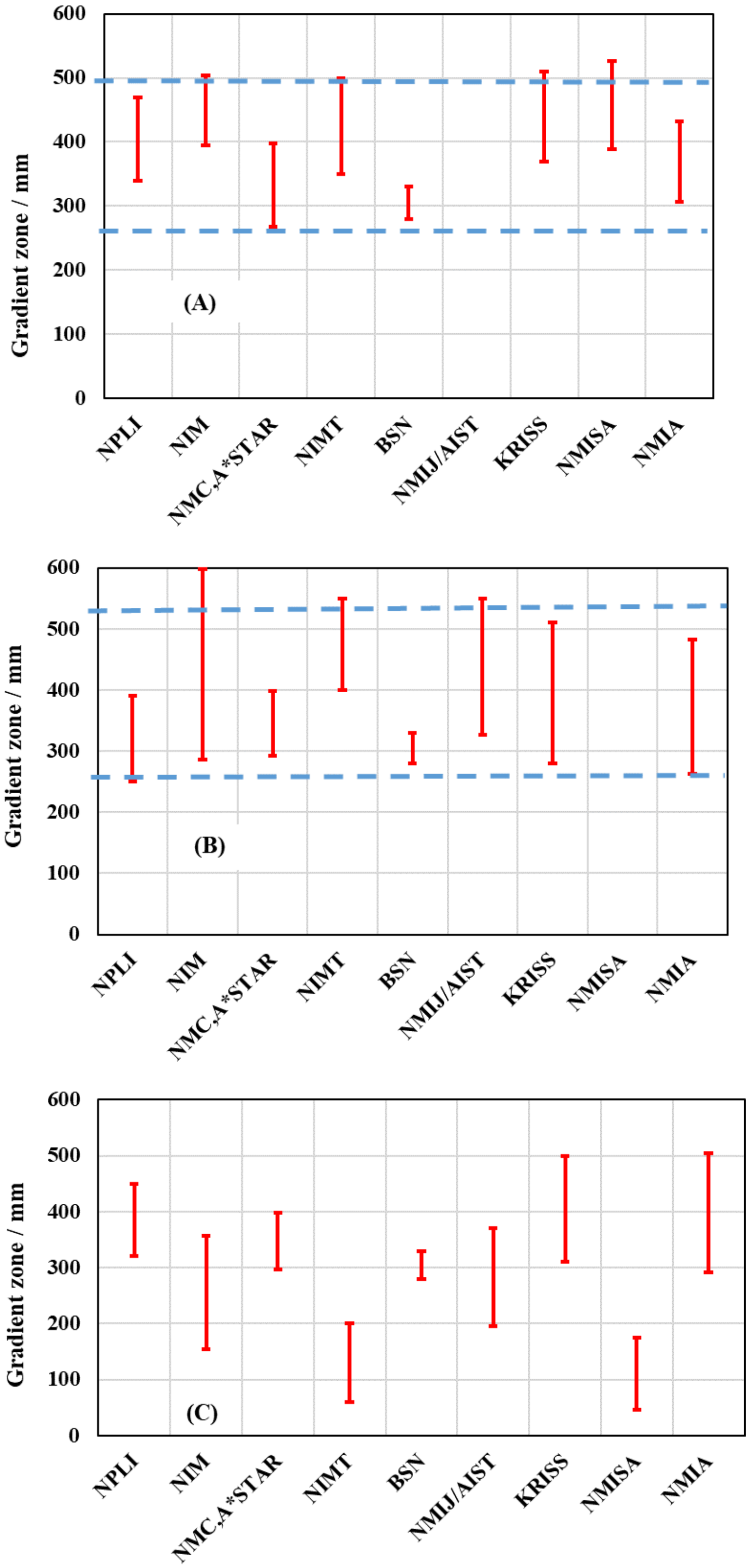


Figure 5. Thermal gradient zone of the furnaces used by participants for: (A) Cu point (B) Co-C point and (C) Pd point.

10.2. Drift of Thermocouple during the Comparison

Each participant calibrated the thermocouple twice to link them to the pilot: at the start and the end of the comparison. Table 9 (based on Tables 2, 3 and 4), gives the value of this drift, which is the difference between the initial and final $E\text{-}E_{ref}$ values. The semi-range of this drift is taken as an additional uncertainty term in the link between the participant and pilot. As the lab, BSN, has not supplied the final measurement value at Pd point, the average drift at Pd points of all TCs, is used for the BSN thermocouple.

Table 9. Difference between final and initial calibrations made by each participant.

Lab Name	Drift of TC, $E\text{-}E_{ref}$ (final-Initial) /µV		
	at Cu Point	at Co-C Point	at Pd Point
NPLI	4.11	6.77	4.15
NIM	0.27	1.30	2.90
NMC, A*STAR	1.55	2.13	0.52
NIMT	0.20	0.01	1.90
BSN	1.60	1.00	*2.41
NMIJ/AIST	-	0.10	0.40
KRISS	0.14	0.70	3.50
NMISA	0.01	-	3.47

* BSN estimated value of drift.

10.3. Calculation of Lab-Pilot value and it's Uncertainty

The analysis and calculation of the comparison reference value is performed in the same manner for each of the three temperature points (Cu, Co-C, and Pd).

The *difference between the participant and pilot laboratory,* X_i, and its associated uncertainty $u(X_i)$ is calculated using the following equations.

$$X_i = \begin{cases} (E-E_{ref})_i - (E-E_{ref})_{pilot} & \text{(for participant i)} \\ 0 & \text{(for the pilot)} \end{cases} \quad (2)$$

and

$$u(X_i)^2 = \begin{cases} (U_i/2)^2 + (U_{pilot,uncorr}/2)^2 + (drift/2)^2/3 + ((E_{ref}(T)-E_{ref}(21)).inhomo_i/2)^2 & \text{(for participant. i)} \\ (U_{pilot}/2)^2 & \text{(for the pilot)} \end{cases} \quad (3)$$

Where

$(E\text{-}E_{ref})_i = [(E\text{-}E_{ref})_{i,initial} + (E\text{-}E_{ref})_{i,final}] / 2$
is the average of the initial $(E\text{-}E_{ref})_{i,initial}$ and final $(E\text{-}E_{ref})_{i,final}$ participant values … from tables 2, 3 and 4

$U_i^2 = (U_{i,initial}^2 + U_{i,final}^2)/2$ average of uncertainty supplied by each lab … from tables 2, 3 and 4.

$E_{ref}(T)$ is the reference emf at the measured temperature of T

$E_{ref}(21)$ is the reference emf at 21°*C.*

$inhomo_i$ is the inhomogeneity of the TC between the pilot and participant furnace … from table 8

$drift_i$ is the drift in thermocouple calibration = $|(E\text{-}E_{ref})_{i,initial} - (E\text{-}E_{ref})_{i,final}|$ … from table 9

U_{pilot} is the reported uncertainty of the pilot lab calibration uncertainty (k=2) … from table 2

$U_{pilot,uncorr}$ is the uncorrelated component of the pilot lab calibration uncertainty (k=2) … from table 2

Note that $X_i=0$ for the pilot, as difference from the pilot to itself is zero by definition, and the uncertainty of the lab-pilot values for the participant have some additional terms corresponding to the drift and inhomogeneity uncertainty from the thermocouple they used to determine the lab-pilot difference. The pilot lab has the special position of direct access to the reference value (RV): as the reference value is expressed in terms of RV-pilot.

The differences between the participants results and the pilot laboratory, X_i , as calculated using equation (2) at Cu, Co-C and Pd temperature points are given in table 10 and plotted in Figure 6 A, B and C respectively. The figures also include the three comparison reference values (calculated in section 11) for each of the three comparison temperatures.

Table 10. **Calculated X_i values at Cu, Co-C and Pd points in μV**

Lab Name	Cu	Co-C	Pd
NPLI	-1.24	4.12	2.95
NIM	1.86	3.97	-0.65
NMC, A*STAR	3.65	5.66	8.83
NIMT	0.10	-2.85	-1.05
BSN	-4.63	-6.03	-23.70
NMIJ/AIST	-	0.30	-6.10
KRISS	2.51	0.30	-1.95
NMISA	-0.80	-	-1.16

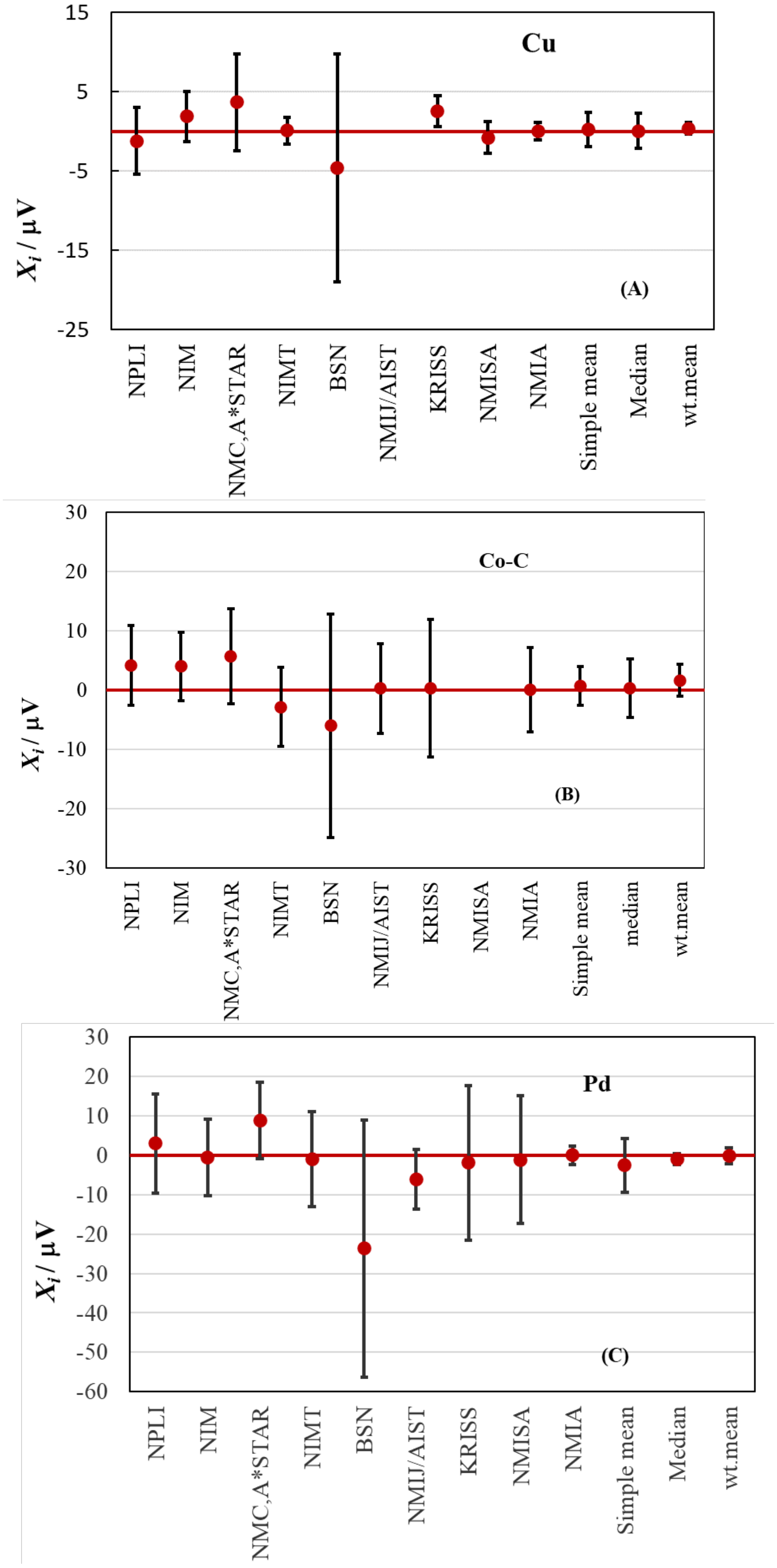


Figure 6. Difference of *E-E_{ref}*, X_i, between each participant and the pilot at Cu (A), Co-C (B) and Pd point temperature (C), together with the calculated values of simple mean, Median and weighted mean.

11 CALCULATION OF COMPARISON REFERENCE VALUE

For each of the three comparison temperatures points, a reference value (RV) X_{RV}, is calculated based on the 9 values of X_i (or 8 for Cu and Co-C). It is important to note that X_{RV} is referenced to the pilot: i.e. it represents the difference of the pilot to the comparison reference value.

The comparison reference values for each of the three temperature points were calculated by three different methods: Simple mean, Median, and Weighted mean, using the three equations below:

i) **Simple Mean:**

$$X_{simple} = \sum X_i / n \quad (4)$$

$$u(X_{simple}) = STDEV(X_i) / \sqrt{n} \quad (5)$$

The simple mean is a good basic estimator of an average; however it does not directly incorporate any of the $u(X_i)$ information, weighting all data equally, regardless of their uncertainties.

ii) **Median:** Computed using the MEDIAN function on Microsoft EXCEL. The uncertainty was calculated using equation given in reference [5]

$$X_{median} = median\{x_i\} \quad (6)$$

$$u(X_{median}) \cong \frac{1.9}{\sqrt{n-1}} median\{|X_{median} - X_i|\} \quad (7)$$

The median generally offers the poorest uncertainty, as it does not directly incorporate all of the information in the X_i and does not use any of the $u(X_i)$ information.

iii) **Weighted mean:**

$$X_{weighted} = \sum X_i . u(X_i)^{-2} / \sum u(X_i)^{-2} \quad (8)$$

$$u(X_{weighted})^2 = 1 / \sum u(X_i)^{-2} \quad (9)$$

The weighted mean generally offers the lowest uncertainty, as it directly incorporates all of the available data and uncertainty information However, the use of the weighted mean requires that the dispersion of the measured X_i are adequately described by their individual estimated uncertainties $u(X_i)$. The Birge ratio [10] and Birge criterion [10], which is a statistical measure of how well the estimated measurement uncertainties explain the measured dispersion of the actual data values, is given by,

$$\text{Birge Ratio} = \sqrt{[\sum (X_i - X_{weighted})^2 u(X_i)^{-2} / (n-1)]} \quad (10)$$

$$\text{Birge Criterion: Birge Ratio} < \sqrt{[1 + \sqrt{(8/(n-1))}]} \quad (11)$$

which is **1.41** for n=9 (Pd point) laboratories and **1.44** for n=8 (Cu, Co-C points) For the comparison here the Birge criterion (11) is well satisfied for all three comparison temperature points (Table 11).

The three different reference values are all consistent within their uncertainties. As the weighted mean provides (a) the overall lowest uncertainty, (b) incorporates participants uncertainty estimates and (c) is statistically consistent with these uncertainties, it is chosen to provide the reference values for the comparison.

Table 11. Calculated Reference Values, their associated expanded uncertainties (k=2, except for simple mean where student t-distribution was used) and calculated Birge Ratio for the weighted mean.

Temperature/°C	Simple Mean / µV		Median / µV		Weighted Mean / µV		
	X_{ref}	$U(X_{ref})$	X_{ref}	$U(X_{ref})$	X_{ref}	$U(X_{ref})$	Birge Ratio
Cu (1084.62 °C)	0.18	2.15	0.05	2.22	0.36	0.73	1.18
Co-C (1324 °C)	0.68	3.25	0.30	4.90	1.62	2.69	0.85
Pd (1554.8 °C)	-2.54	6.84	-1.05	1.41	-0.16	2.05	1.02

The uncertainty of the reference values achieved in the comparison were excellent, corresponding to:

±0.06 °C (k=2) at Cu point (1084.62 °C)
±0.19 °C (k=2) at Co-C point (1324 °C), and
± 0.15 °C (k=2) at Pd point (1554.8 °C).

It is interesting to note that the RV uncertainty achieved in APMP.T-S1-04 at 1084 °C was also close to 0.06 °C.

12 DEVIATION FROM THE REFERENCE VALUE

The degree of equivalence D_i of the participating lab with respect to the comparison reference value and its uncertainty u_{Di} are calculated from the following equations :

$$D_i = X_i - X_{weighted} \qquad (11)$$

$$u_{Di}^2 = u(X_i)^2 + u(X_{weighted})^2 \qquad (12)$$

Note: $u(X_i)$ is the value calculated using equation 3.

The En number is calculated for each participant, including pilot, using the following formula

$$En = |D_i|/U_{Di} \qquad (13)$$

The values of D_i, U_{Di} (k=2) and the En number for the participants and pilot are given in table 12 and presented graphically in figures 7 (A, B, and C). All participants are seen to be in good agreement with the reference values.

Note: The raw U_{95} uncertainties provided by each participant are also plotted in the same plots (these are the smaller error bars in the graph), to illustrate that the four additional terms from the drift, inhomogeneity, uncorrelated pilot lab uncertainty and weighted uncertainty have not significantly increased the uncertainties from each lab. The comparison can thus be considered to be a good test of the capabilities of each lab.

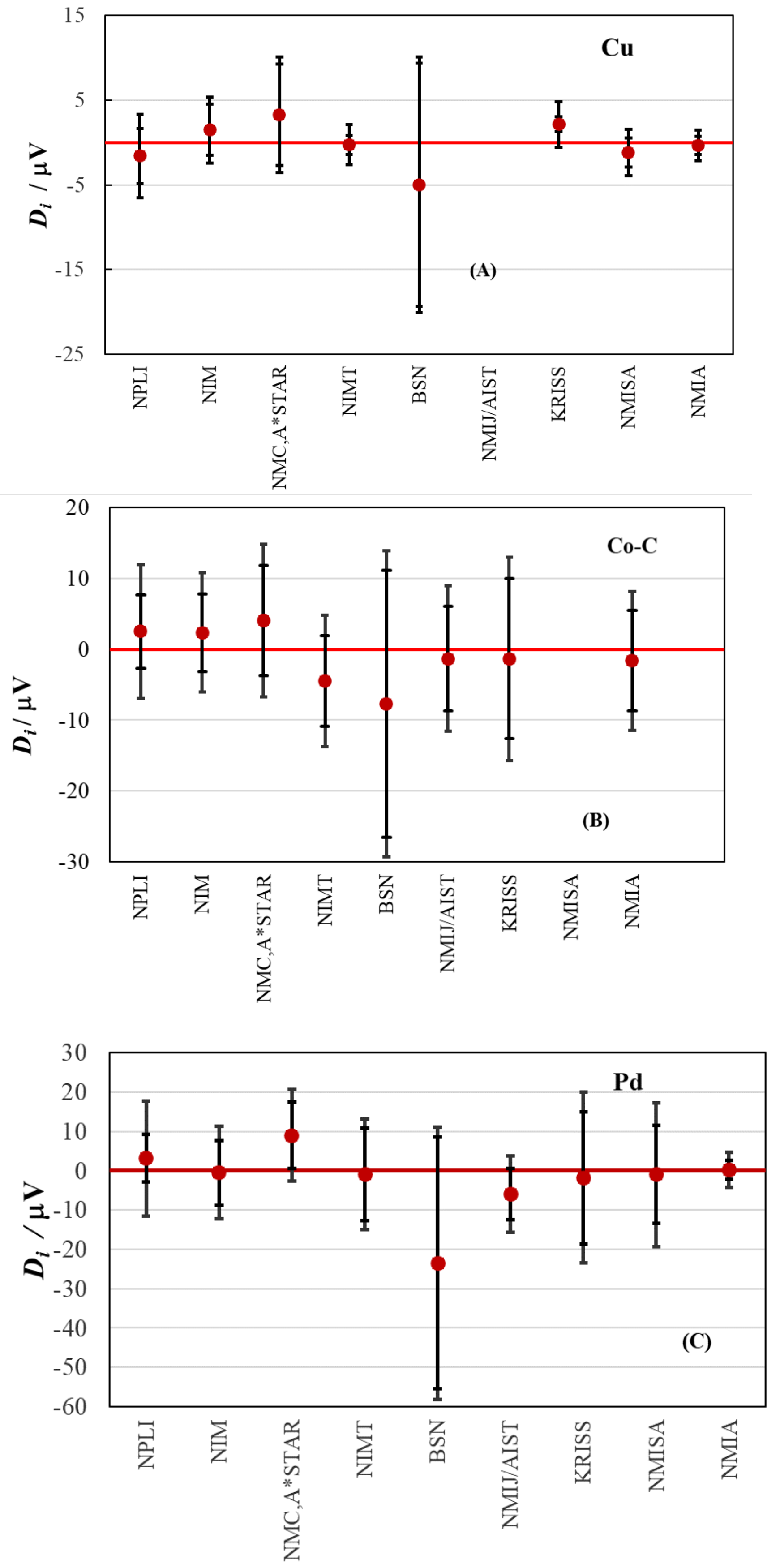


Figure 7. The degree of equivalence of each participant and pilot, D_i; at (A) Cu point (B) Co-C point and (C) Pd point. The smaller error bars are the uncertainties provided by each participant.

Table 12. Calculated Values of Degree of equivalence D_i, the expanded uncertainties U_{Di} (k=2) in μV and the *En* number for all participants.

Lab.	Cu Point			Co-C Point			Pd Point		
	D_i	U_{Di}	*En*	D_i	U_{Di}	*En*	D_i	U_{Di}	*En*
NPLI	-1.6	5.0	0.3	2.5	9.4	0.3	3.1	14.7	0.2
NIM	1.5	3.9	0.4	2.3	8.4	0.3	-0.5	11.8	0.0
NMC,A*STAR	3.3	6.9	0.5	4.0	10.7	0.4	9.0	11.7	0.8
NIMT	-0.3	2.4	0.1	-4.5	9.3	0.5	-0.9	14.1	0.1
BSN	-5.0	15.1	0.3	-7.7	21.6	0.4	-23.5	34.7	0.7
NMIJ/AIST				-1.3	10.3	0.1	-5.9	9.7	0.6
KRISS	2.2	2.7	0.8	-1.3	14.3	0.1	-1.8	21.7	0.1
NMISA	-1.2	2.8	0.4				-1.0	18.2	0.1
NMIA	-0.4	1.8	0.2	-1.6	9.8	0.2	0.2	4.4	0.04

13 CONCLUSION

An inverted star type comparison was conducted among 9 APMP NMIs (including pilot) for the calibration of type R thermocouples at the Cu, Co-C and Pd temperature points.
By specifying the annealing state and considering the inhomogeneity values only over the difference in furnace gradient zone between pilot and participant the uncertainty due to drift and inhomogeneity could be minimised. Despite the wide variation in calibration techniques used by the 9 laboratories, this allowed the determination of comparison reference values with low uncertainties (comparable to that of ITS-90),

±0.06 °C (k=2) at Cu point (1084.62 °C)
±0.19 °C (k=2) at Co-C point (1324 °C), and
± 0.15 °C (k=2) at Pd point (1554.8 °C).

This low uncertainty in reference value allowed achieving participants' degrees-of-equivalence comparable to their claimed uncertainty. The results also showed *En* ratios <1 at all comparison temperatures for all participants. This supports the equivalence between the wide range of calibration references and methods adopted (eg. Pd fixed point cell, Pd melting point in air, Pd melting point in argon and comparison).

14 REFERENCES

1. F. Jahan & M. Ballico, "Final report on the APMP Intercomparison of Type R Thermocouples Calibration from 0 to 1100 °C", APMP-T-S1, Metrologia, **2007**, vol 44, Tech. Suppl., 03004
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APPENDIX A: Protocol of the comparison (APMP.T-S16)

Protocol for the APMP Regional Comparison of Type R Thermocouples Calibration

(version 6)

APMP.T-S16

June 2017

F Jahan
National Measurement Institute of Australia

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1. INTRODUCTION

In 2007 an APMP regional comparison of the calibration of rare metal (Pt-Pt13%Rh) thermocouples from 0 to 1100 °C was successfully ran by the National Measurement Institute of Australia and is partially funded by APEC (Asia Pacific Economic Cooperation). Twelve laboratories of the Asia Pacific region took part in the comparison, which was published in Metrologia [1].

Pt based thermocouples are widely used to provide traceability to accredited laboratories at high temperature (>1000 °C), and are routinely calibrated by most of the regional NMI within APMP up to 1560 °C. Although a successful comparison to 1100 °C (APMP.T-S1-04) has been performed, no comparison has so far been made over the higher temperature range.

In general, a thermocouple calibration consists of measuring the thermocouple emf, E, at several values of tip temperature and comparing the emf with the corresponding value, E_{ref}, given by a defined reference function [2]. Different NMIs have adopted different procedures to calibrate thermocouples in the temperature range (1100 °C to 1560 °C): such as Au and Pd 'melt wire' techniques (in both air and argon), mini fixed point of Cu, and Co-C and Pd-C eutectic points, and comparison with reference thermocouples and/or radiation thermometers. ***A key function of this comparison is to examine the equivalence of the diverse techniques used by the APMP NMIs for the calibration of client's Pt-based thermocouples over 1100-1560*** °C.

The pilot lab (NMIA) has surveyed the facilities used by the APMP laboratories in this high temperature range for the calibration of Pt-based thermocouples to help guide the design of the comparison. Nine potential participants responded: 7 use Au and Pd-melt wire techniques and Co-C eutectic fixed points; 8 use a Cu fixed point; four labs have facilities of using the Pd-C eutectic point.

Key issues considered in the design of the protocol were:

- The need to conduct the comparison quickly.
- The need to minimize the work load to the pilot laboratory.
- The need to achieve the best accuracy for the comparison to support CMCs in the range 1100-1560°C.
- The need to minimize the risk of thermocouple drift and increased inhomogeneity due to contamination, heat treatment and also change of length of the wires.

Considering these factors, we have decided that:

- Each laboratory will calibrate the thermocouple at Cu (optional), Co-C and Pd melting point or by comparison at these temperatures using their own test methods. The Pd-C point was not considered as there is the risk of significant drift of thermocouple [3].
- An inverted-star type comparison will be used, to make the intercomparison process quicker.
- The inhomogeneity of thermocouples will be measured at several stages by the pilot lab, NMIA to allow for uncertainty due to differing furnace gradients of the participants.
- The pilot laboratory, NMIA (Australia) will construct the 8 type R thermocouples; each participating laboratory will receive one thermocouple in the 1100 °C quenched state.

Nine laboratories of the Asia Pacific region will take part in this comparison including pilot lab NMIA. The names of the laboratories are given in **Appendix A,** and the methods and equipment reported as required in **Appendix B.** The calibration results will then be sent to the pilot laboratory in the format given in the **Appendix C**. The results of the calibration from different laboratories will be analyzed by NMIA, who will prepare the draft-A and B reports on the intercomparison.

2. DESCRIPTION OF THE THERMOCOUPLES

Platinum-Platinum 13%Rhodium (Type R) reference grade thermocouple made from same batch of wires from Sigmund Cohn, USA. The wire length is approximately 1400 mm and is installed in a 4.1 mm diameter high purity alumina insulator which has been prebaked at 1150 °C. Approximately 700 mm of thermocouple wire emerging from the alumina tube and are insulated with PVC sleeves. The serial number of the thermocouples are as follows with the name of the laboratory:

i) 2017-02 – NPLI, India
ii) 2017-05 – NIM, China
iii) 2017-06 – NMC, A*STAR, Singapore
iv) 2017-08 – RCM-LIPI, Indonesia
v) 2017-09 – NMIJ, Japan
vi) 2017-10 – KRISS, Korea
vii) 2017-11 – NMISA, South Africa
viii) 2017-07 – NIMT, Thailand

3. MEASUREMENT SEQUENCE IN THE COMPARISON:

The comparison will be conducted as an inverted star i.e.
Participant → Pilot → Participant

- **March 2017:** Manufacture of 8 thermocouples by the pilot laboratory, NMIA
- **March - April 2017:** Annealing at 1100 °C and measurement of the inhomogeneity of 8 thermocouples at 200 °C by the pilot laboratory, NMIA,
- **April 2017:** Send thermocouples to the participating laboratories
- **April - June 2017:** Calibration of the thermocouples by the participating laboratories (initial measurement, round #1)
- **July 2017 :** Thermocouples returned to the pilot laboratory, NMIA
- **July – September 2017**: Pilot will anneal thermocouples at 1100 °C, then Measurement of the inhomogeneity of 8 thermocouples at 200 °C and calibration of 8 thermocouples by the pilot laboratory.
- **October 2017:** Pilot to anneal thermocouples at 1100 °C and send back to the participants..
- **November – December 2017:** Calibration by the participating lab as before (Final measurement, round #2)
- **January – 2018:** Send back the Thermocouple to NMIA to measure the inhomogeneity (**Optional:** as depending on the values of round #1 and round #2. If the two values of EMF of initial and final calibration agreed to each other within the claimed uncertainty of the participating laboratory, then no need to measure inhomogeneity again. For example, if U_{95} ($k = 2$) is 1.5 μV, at Cu point, then the value of initial and final values of EMF should agree within 1 μV).

4. INSTRUCTIONS TO THE PARTICIPATING LABORATORY:

- Each participating laboratory will receive one thermocouple, which is in ‘‘1100 °C Quenched state”, that is, annealed at 1100 °C for 1 hour and quenched.
- Upon receiving the thermocouple, the participating laboratory must inspect the thermocouple for any damage and report to NMIA, if any damage is detected. NMIA will give instructions how to proceed.
- Measure and record the length of the thermocouple wires.
- Connect a pair of Cu- wires to the CJ (cold-junction end or open end) of the thermocouple.
- Calibrate the thermocouple at the fixed points of Cu, Co-C, and Pd melting point, or by comparison techniques, using the normal calibration technique as practiced by the participant laboratory for their clients.
- The calibration sequence should be from **lower to higher** temperatures.
- Participants should minimize the length of time the thermocouple is exposed to temperatures above 1000 °C.
- Participants must record and report the time the thermocouple is exposed to temperature above 1000 °C.

- The immersion depth of the thermocouple in the calibration enclosure should be **600 mm or less** from the tip.
- Measure the temperature uniformity of the enclosure used during calibration and measure the gradient zone of the furnaces as shown in Figure 1 (to provide the immersion length of the thermocouple as in Table in Appendix B.
- After completion of the calibration, measure and record the length of the thermocouple wires.
- The participant laboratory should transfer the data and the thermocouple to NMIA.

Important Notes:

i) The participating laboratory **should not dismantle** the thermocouple.
ii) Each fixed point should be realized **3 times**.
ii) Please **avoid unnecessary measurements** to reduce the drift at higher temperature**.**
iii) In case of **Pd melt wire technique**, please make sure that Thermocouple wires **are not shortened by more than 15 mm to 20 mm in total (i.e <5mm per melting point run)**
iv) If using different enclosures for different fixed point, if possible, the thermocouple may be annealed at 1100 °C for 45 minutes and quenched before changing the enclosure (to minimize inhomogeneity errors).
v) If a participant fails to submit the results by the due date (except for special reasons such as failure of artifacts), the participant will be disqualified.

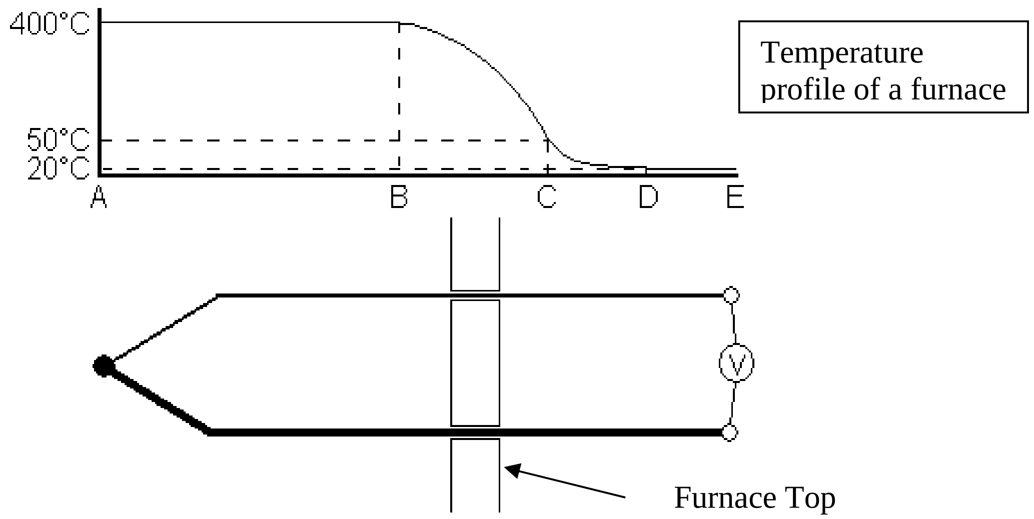

Figure 1. Schematic diagram showing the temperature gradient and immersion of thermocouple. Please provide the length of A to B and A to C, A is the position of the tip of the thermocouple, B and C are the positions where temperature of the furnace, T drops to 90% and 10%.

5. REPORTING DATA TO NMIA:

The participating laboratory must send to NMIA the following information **within 12 weeks** of receiving the thermocouple:

1. A general outline of the calibration procedure consisting of no more than one page and send this as an electronic file named **‘procedure.doc’**, including a description, how the temperature of a fixed point (Co-C fixed point) was determined.

2. Details of instrumentation used in the comparison calibration as an attached Excel spreadsheet named ‘**Instrument.xls’ as in Appendix B. Please provide the temperature gradient zone of the furnaces used during measurement** in accordance to figure 1 shown in **Appendix B**.

3. The values of calibration results as an Excel spreadsheet named **‘Calibrationdata.xls’.** This should be in the format given in **Appendix C.** The results of initial measurements (round #1) should be delivered to the pilot lab, before receiving the thermocouple for final measurement (round #2).
4. The uncertainty analysis according to the ‘ISO Guide to the expression of Uncertainty in Measurement’ in terms of microvolt. Please send an electronic file named **‘Uncertainty.xls’**. The terms in the **Appendix D** should be used as a guide. Individual laboratory may add any additional uncertainties if they consider relevant. Please send a brief description on how the each component used are evaluated, like as in Appendix D.

   Note: Use the inhomogeneity value of the test thermocouple as given by the NMIA with the thermocouple.

6. TRANSPORTATION OF THERMOCOUPLES:

- The pilot laboratory, NMIA will send a thermocouple in its wooden box door-to-door to each participating laboratory.
- It is the responsibility of each laboratory to arrange transport of the thermocouple after calibration, back to the pilot laboratory.
- It is the responsibility of each laboratory to obtain insurance for the artifacts for transport.
- The instrument should be accompanied by an ATA carnet (if needed) or customs declaration document and Received/Dispatched form as given in **Appendix E.**
- The participating laboratory is responsible for all expenses related to the transport of the thermocouple to and from the lab.

7. ANALYSIS OF DATA

The proposed method of analysis will follow that used for APMP.T-S-01-04, and reported in [1, 4]

The pilot laboratory will combine the uncertainty analyses provided by the participants with the uncertainty due to thermocouple inhomogeneity in the calculation of the final participant uncertainty. This will include the measured inhomogeneity data for each thermocouple, and nominal furnace gradient zone for each furnace, as provided by the participants. The difference between the initial and final measurements by each participant will be used to calculate an additional artefact-stability uncertainty, if there is statistically significant evidence of any drift, given the reported participant uncertainty. The difference between each participant and the pilot laboratory will be calculated, and a reference value for each comparison temperature calculated based on the simple mean, weighted mean and median of these differences. The choice of which average to use will be made after the release of the draft-A and examination of the data.

References:

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14.1 Appendix A: List of Participating Laboratories

Name of Laboratory	**Contact Person**	**Address**
NMIA – Australia	Ferdouse Jahan ferdouse.jahan@measurement.gov.au	36 Bradfield Rd, West Lindfield NSW 2070, Australia
NPLI – India 2017-02	D. D. Shivagan shivagand@nplindia.org	CSIR-National Physical Laboratory, Dr. K. S. Krishnan Marg, New Delhi 110012, India.
NIM – China **2017-05**	Zheng wei zhengw@nim.ac.cn	National Institute of Metrology Division of thermophysics and process measurements. No.18, Beisanhuan Donglu, Beijing, China 100013
NMC, A*STAR – Singapore **2017-06**	Fan Yan fan_yan@nmc.a-star.edu.sg	National Metrology Centre 1 Science Park Drive PSB Building, Singapore 118221
RCM-LIPI – Indonesia **2017-08**	Beni adi Trisna beni.ugm05@gmail.com	Kompleks PUSPIPTEK Gd. 420, Serpong, Tangerang Selatan 15314, Banten, Indonesia.
NMIJ – Japan **2017-09**	Hideki Ogura h.ogura@aist.go.jp	AIST, Central 3, 1-1-1 Umezono, Tsukuba, Ibaraki 305-8563, Japan
KRISS- Korea **2017-10**	Kim Yong-Gyoo dragon@kriss.re.kr	267 Gajeong-ro, Yuseong-Gu, Daejeon 34113, Korea CP: 010-3401-6334
NMISA – South Africa **2017-11**	Efrem Ejigu EEjigu@nmisa.org	NMISA – CSIR Campus Meiring Naude Road, Brummeria ZA-0001,Pretoria,South Africa
NIMT – Thailand **2017-07**	Oijai Ongrai oijai@nimt.or.th	National Institute of Metrology (Thailand) 3/4-5 Moo 3, Klong 5, Klong Luang, Pathumthani 12120

14.2 Appendix B: Measuring equipment used in the comparison.

Laboratory Name: ______________________________

Length of thermocouple wires: i) As received __________ mm
ii) After measurement __________ mm
iii) Number of hours >1000°C __________

Devices	**Type**	**Manufacturer**	**Serial number**	**Description**	**position of temperature gradient zone (mm)**	
					A-B (tip to 90% T)	**A-C (tip to 10% T)**
Ice-Point used						
DVM used						
Scanner (if used)						
Fixed Point /Enclosure used	Cu point					
Fixed points /Enclosure used	Co-C point					
Fixed points / Enclosure used	Pd point					
Other enclosure						

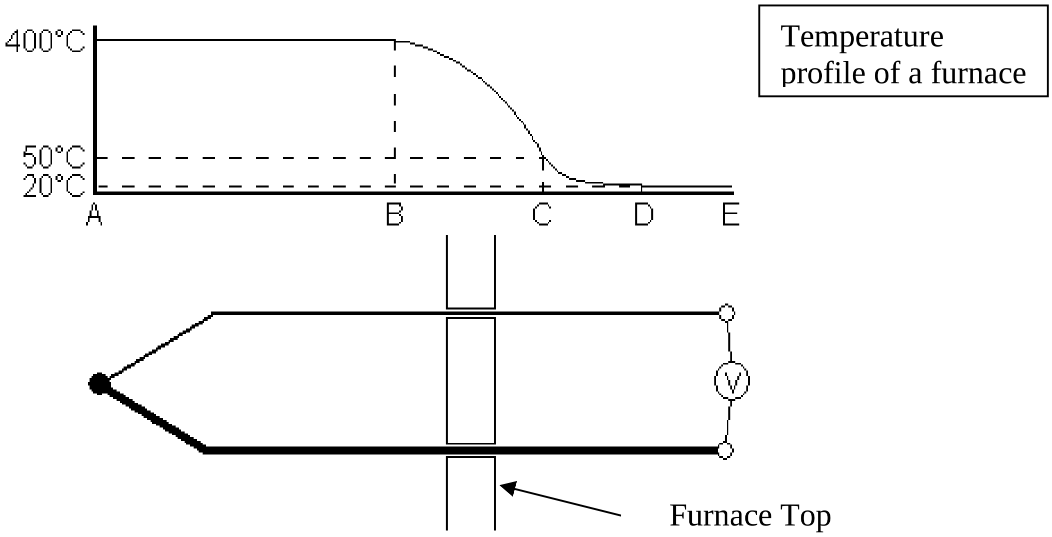


Figure 1. Schematic diagram showing the temperature gradient and immersion of thermocouple. Please provide the length of A to B and A to C, A is the position of the tip of the thermocouple, B and C are the positions where temperature of the furnace, T drops to 90% and 10%.

Appendix C: Calibration Data of the Thermocouple

Serial Number _______________ Inhomogeneity (from NMIA)________________

Name of the Laboratory:____

Temperature (nominal T)	**T_{ref} /°C** eg. after corrections or cell calibration	**E_{ref}/ μV** calculated from T_{ref}	**E_{meas} /μV**	**E_{meas} - E_{ref} /μV**	**Average E-E_{ref}/μV**	**Uncertainty (*k*=2) /μV**
1084 °C (eg. Cu point)			i) ii) iii)	i) ii) iii)		
1324 °C) (eg. Co-C point)			i) ii) iii)	i) ii) iii)		
1554 °C (eg. Pd (point)			i) ii) iii)	i) ii) iii)		

Appendix D : Uncertainty Analysis

The participating laboratory should send the calculated uncertainty of measurement to the pilot laboratory, as an Excel spreadsheet named **'Uncertainty.xls'.** To calculate the uncertainty of calibration, the participants should follow the guideline set out in the 'ISO Guide to the Expression of Uncertainty in Measurement'. The various uncertainty components are given below as a guide:

- **Uncertainty of the Fixed point temperature**: an uncertainty value is assigned to the particular fixed point to cover the purity of the fixed point metal, (which can be estimated by melting range of the fixed point, Melt/freeze agreement, flatness of the freezing or melting curve) and also factors related to the realisation of the fixed point such as the choice of position on the freezing plateau.
  NOTE: if comparison techniques (eg a reference thermocouple or a radiation thermometer are used), put the uncertainty in the assigned reference temperature here
- **Measurement scatter at the fixed point:** This is a type-A component. The reproducibility of EMF measurements in the fixed point. The standard deviation of 3 freezing point values should be used.
- **Conduction errors:** This will typically be estimated by pulling the thermocouple should be pulled out by 1 to 2 cm from full immersion and observing the change in measured thermocouple EMF. However, each laboratory will have their own techniques, and should specify in their reported methodology how they estimated the numerical value they assigned here.
- **Uncertainty due to Inhomogeneity of thermocouple: "Exclude this component"** The inhomogeneity in Seebeck coefficient along the thermocouple being calibrated would be measured by the pilot lab. The inhomogeneity component will be added by the pilot lab during analysis of the results from the reporting gradient zone/ immersion length of the thermocouples.
- **CJ temperature:** This component is estimated from the quality of the ice point and also depends on the immersion and slight inhomogeneity (if any) of this section of the thermocouples.
- **DVM calibration and its use**: This is calibration uncertainty of the DVM used and the drift of the DVM during use since calibration.
- **Rounding/ Resolution error**: The rounding error or the resolution of the reported EMF at each of the fixed point.
- **Stray EMFs and electrical Noise**: Any spurious EMFs caused by AC pickup during measurement at the fixed points.

Appendix E: i) Customs Declaration

TO WHOME IT MAY CONCERN

APMP Regional Comparison

The Asia Pacific Metrology Program (APMP) is an organisation representing the National Measurement/ Standards Laboratories of a large number of countries/territories in the Asia-Pacific region. Its broad objective is to improve the measurement capabilities in the Asia-Pacific region by sharing facilities and experience in metrology.

One very successful method used by the APMP is the comparison of calibrations performed by different laboratories on a given artefact. Successful completion of these intercomparisons adds confidence to the laboratories in the carrying out of standards measurements and leads to international acceptance of the measurements carried out by these laboratories.

As part of a major intercomparison program, the APMP is conducting an intercomparison on the calibration of type R thermocouple from 1100 °C to 1560 °C involving the participants given in Appendix A.
This program is coordinated by,
National Measurement Institute of Australia
Lindfield, NSW 2070
Australia

The following artefact is circulated among the participants for calibration:

A type R Thermocouple, Serial number:

The purchase/manufacturing cost of the artifact was AUS$1500. However it has no commercial value (it is not for sale). It is meant solely for the calibration of national standards and will be re-exported immediately after the calibration is complete (see enclosed Schedule).

We request that the device is not handled or removed from the container/package. If a Customs inspection is required then please contact the relevant person listed in the attached schedule so that he/she can be present and help you unpack it.

Comparison Co-coordinator
Dr. Ferdouse Jahan
NMI, Australia

Appendix E: ii) Received/Dispatched Form

B) ARTEFACT RECEIVED

To:…*(sender / coordinator)*….

APMP Regional Key Comparison APMP.T-S16

The …*(artifact)*…. and its ATA Carnet was received at ……..*(name of laboratory)*…. on …*(date)*..

The condition when it was received was *in good physical and working order

*damaged – *(explain)*

____
(Name of participant)

C) ARTEFACT SHIPPED

To: *(recipient / coordinator)*

15 APMP Regional Key Comparison APMP.T-S16

The …*(artifact)*…. and its ATA Carnet was hand delivered to………*(name of person)*….. at …*(name of laboratory)*..….. on …….*(date)*……

____
(Name of Participant)

APPENDIX B: Instrument used by the Participants

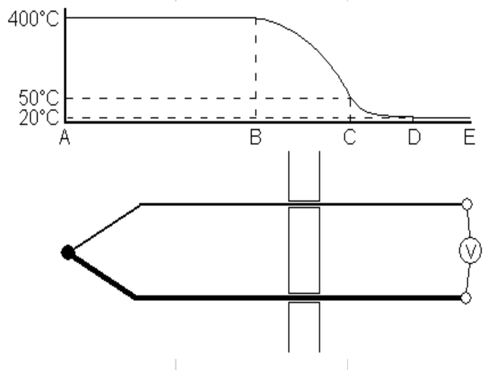
B-1) NMC, A*STAR

Measuring equipment used in the comparison.

Laboratory Name: **Temperature and Humidity Laboratory, National Metrology Centre, A*STAR**

Length of thermocouple wires: i) As received 1350 mm
ii) After measurei 1350 mm
iii) Number of hours >1000°C 46 hours 25 min

Devices	Type	Manufacturer	Serial number	Description	position of temperature gradient zone (mm)		Remarks
					A-B (tip to 90% T)	A-C (tip to 10% T)	
Ice-Point used	Self prepared ice point			Melting point of ice prepared according to procedure TS/IP/002, issue no 2.			General equipment used
DVM used	Nanovoltmeter Model: 2182A	KEITHLEY	1238213	100 mV range, accuracy is ± (30*ppm of reading + 4*ppm of range)			
Scanner (if used)	Low Thermal Scanner Model:160B	DATA PROOF	1244	Input channel 1 is used for thermocouple			
Fixed Point used	Cu fixed Temperature point	ISOTECH	CU 85	The slim Cu sealed cell encased in quartz, was Melt/Freeze in a Oberon	247 mm	342 mm	Direct Fixed Point Method
Enclosure used	1084.62 °C Temperature point	GERO Hochtemperat urofen GMBH & CO.KG	20070852	SiC blackbody and protection cone in the furnace tube	268 mm	398mm	Comparison Method
Enclosure used	1324 °C Temperature point	GERO Hochtemperat urofen GMBH & CO.KG	20070852	SiC blackbody and protection cone in the furnace tube	280 mm	398 mm	
Enclosure used	1555 °C Temperature point	GERO Hochtemperat urofen GMBH & CO.KG	20070852	SiC blackbody and protection cone in the furnace tube	290 mm	398 mm	
Other enclosure							



Temperatur

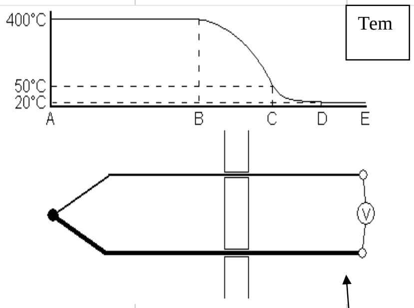
Figure 1. Schematic diagram showing the temperature gradient and immersion of thermocouple. Please provide the length of A to B and A to C, A is the position of the tip of the thermocouple, B and C are the positions where temperature of the furnace, T drops to 90% and 10%.

B-2) BSN

Laboratory Name **<u>: Research Center for Metrology - LIPI (RCM - LIPI)</u>**

Length of thermocouple wires	(i) as received	: 1400 mm
	(ii) after measurement	: 1400 mm
	(iii) Number of hours > 10(	: 27.5 hours

Devices	Type	Manufacturer	Serial Number	Description	Position of temperature gradient zone	
					A-B (tip to 90% *t*)	A-C (tip to 10% *t*)
Ice-point used	7196	Fluke	-	The melting point of ice was realized using this container		
DVM used	2182A	Keithley	1053487	This thermocouple is traceable to SI units through RCM-LIPI's electrical standards		
Standard Thermocouples	(i) Pt/Pd thermocouple	NPL	NPL 07/17/B	This thermocouple is traceable to SI units through NPL's temperature standards		
Furnace	A single-zone-controller horizontal furnace	Land Instrumen	Landcal P1600B	-	28 cm	33 cm

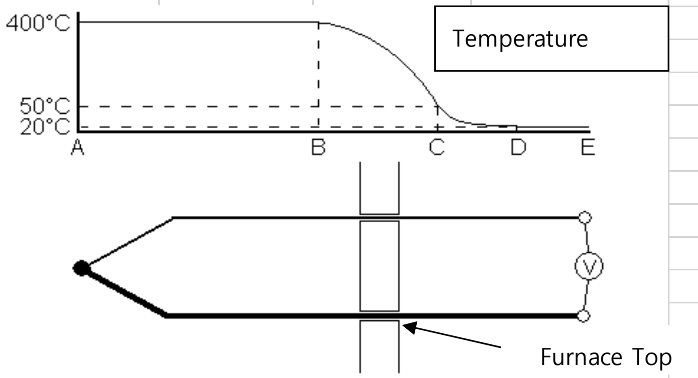


Furnace Top

B-3. KRISS

Laboratory Name:	**KRISS**					
Length of thermocouple wires:		i) As received		1389	mm	
		ii) After measurement		1389	mm	
Number of hours above 1000 °C:		15h 33 min	at first round,		at second round	

Devices	Type	Manufacturer	Serial Number	Description	Position of temperature gradient zone (mm)	
					A-B (tip to 90 % t)	A-C (tip to 10 % t)
Ice-point used	Ice+Water mixture in dewar	KGW	None	Maximum immersion depth: 285 mm		
DVM used	2182A Nanovoltmeter	Keithley	1232368			
Scanner (if used)	Not used	-	-			
Fixed point /Enclosure used	Cu point	KRISS	Cu-O-14-01	Open cell, 3 zone furnace	370	510
Fixed point /Enclosure used	Co-C point	KRISS	CoC-07-01	Open cell, Used in APMP.T-S7 comparison	280	510
Fixed point /Enclosure used	Pd point	KRISS	Pd-16-01	Open cell, Alumina crucible	310	500
Other enclosure						



B-4. NIM

Laboratory Nam	**NIM**					
length of thermocouple wires:		i) As received		1388	mm	
		ii) After measurement		1377	mm	
		iii) Number of hours >1000°(		19		

Device	Type	Manufacturer	Serial Number	Description	position of temperature gradient Zone(mm) A-B (tip to 90%T)	A-C(tip to 10%T)
Ice-Point used	-	NIM	TC-3	Ice-water mixture, maximum 280mm in depth		
DVM used	2182	Keithley	0756530	Range 100mV,Resolution 0.01μV		
Scanner (if usec	160B	Data Proof	1468	Termal Offest Maximum <50nV		
Fixed Point / Enclosure used	Cu Freezing Point/Furnace（9116A）	Hart Scientific	CU 09032 /A89016	Sealed Type Cell /Na Heat Pipe	395	503
Fixed Point / Enclosure used	Co-C HTFP/ Furnace（MAT-60SC2）	NIM/Chino	Co-C #1/070201	Co-C Cell 120mm length,41mm OD/3 zone Furnace	286	598
Fixed Point / Enclosure used	Pd Melting Point/ Furnace	Alfa Aesar /NIM	LOT A26S030/M #2	Purity of Pd wire 99.99+% / Furnace temperature ramp rate was controlled.	154	357
other Enclosure						

B-5. NIMT

1.1 Measuring equipment used in the comparison.

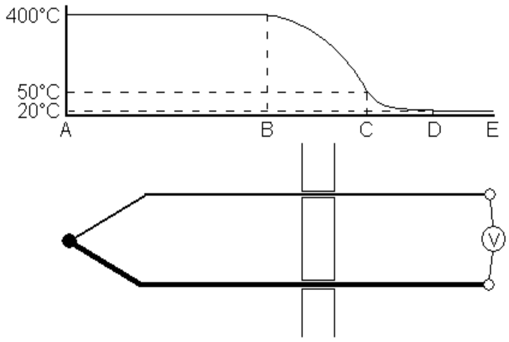
Laboratory Name: National Institute of Metrology (Thailand)

Length of thermocouple wires : i) As received _

ii) After measurement 1385 mm

iii) Number of hours >1000°C 30.5

Devices	Type	Manufacturer	Model	Serial number	Description	position of temperature gradient zone (mm) A-B (tip to 90% T)	position of temperature gradient zone (mm) A-C (tip to 10% T)
Ice-Point used	Cold Junction	Cole parmer	Thermocut - D2000	3763	Vacuum flask filled with mixing of crashed iced and distilled water		
DVM used	Digital Multimeter	Agilent	HP3458		Digital Multimeter		
Scanner (if used)	-	-	-	-	-		
Fixed Point /Enclosure used	Cu point	Isotech / Fluke	3/5/7440/(9116)	/(A6C304)	Sealed cell / three zone furnace	350	500
Fixed points /Enclosure used	Co-C point	NPL, UK / Elite	Co-C / (TMV16/75/610)	(NPL/2014/Co-C1) / (3227/02/14)	Open cell / three zone furnace	400	550
Fixed points / Enclosure used	Pd point	Sigma Aldric	348694 /(TSV18/15/100)	/(3226/02/14)	99.9% Pd wire dia. 0.5 mm / High temp. Furnace	60	200
Other enclosure	furnace	Hart Scientifi	9117	A5B040	Pre-heating furnace		



B6. NMIJ

Measuring equipment used in the comparison.

Laboratory Name: NMIJ

Length of thermocouple wires:
i) As received (+) 2274 mm, (-) 2319 mm
ii) After measurement (+) 2257 mm, (-) 2306 mm
iii) Number of hours >1000 °C 37 hours

Devices	Type	Manufacturer	Serial number	Description	position of temperature gradient zone (mm)	
					A-B (tip to 90% T)	A-C (tip to 10% T)
Ice-point used	D-6000	Thermos	---	Dewar flask. The maximum depth is 250 mm.		
DVM used	8508A	Fluke	939154330	---		
Scanner (if used)	160A Opt.2	Data Proof	845	---		
Fixed point /Enclosure used	Cu point	---	---	---	---	---
Fixed point /Enclosure used	Co-C point	NMIJ /NMIJ	CoC-a26 /MCF-1	--- /3 zone furnace	326	550
Fixed point /Enclosure used	Pd point	Ishifuku Metal Industry Co., Ltd /CHINO Co.	Pd-9 /MAT-70KSVD	Pd wire /3 zone furnace	195	370
Other enclosure	---	---	---	---	---	---

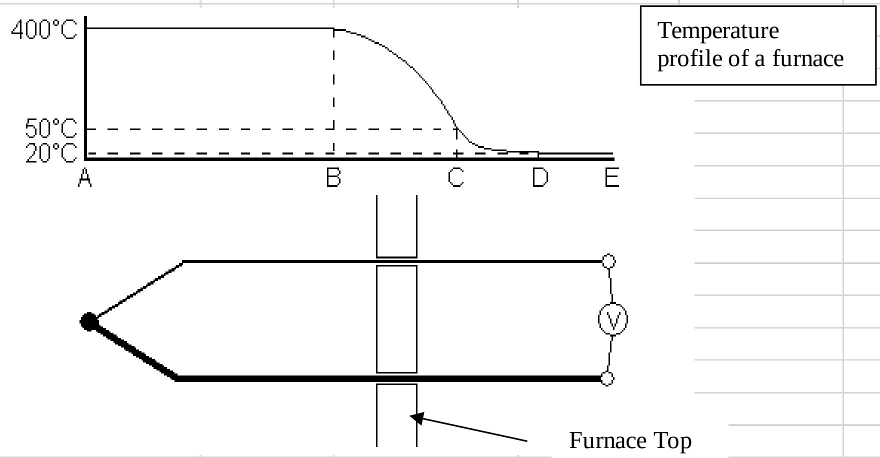


Figure 1. Schematic diagram showing the temperature gradient and immersion of thermocouple. Please provide the length of A to B and A to C, A is the position of the tip of the thermocouple, B and C are the positions where temperature of the furnace, T drops to 90% and 10%.

B-7. NMISA

Appendix B: Measuring equipment used in the comparison.

Laboratory Name: National Metrology Institute of South Africa (NMISA)

Length of thermocouple wires: i) As received 1360 mm
ii) After measurement 1320 mm
iii) Number of hours >1000°C 121 hours

Devices	Type	Manufacturer	Serial number	Description	position of temperature gradient	
					A-B (tip to 90% T)	A-C (tip to 10% T)
Ice-Point used	AF 10	Scotsman	DPT 46252	Ice maker		
DVM used	34420A	Hewlett Packard	US36000373	Nanovoltmeter		
Scanner (if used)	Keithley	705	521077	Scanner		
Fixed Point /Enclosure used	Cu fixed point	Fluke	TS-047 (Cu29056)	Open fixed point cell	389.1	525.9
Fixed points /Enclosure used	Co-C point					
Fixed points / Enclosure used	Pd wire melting furnace	Johnson Matthey	3896 (TE-008)	wire bridge_melt	45.3	174.2
Other enclosure						
Other enclosure						

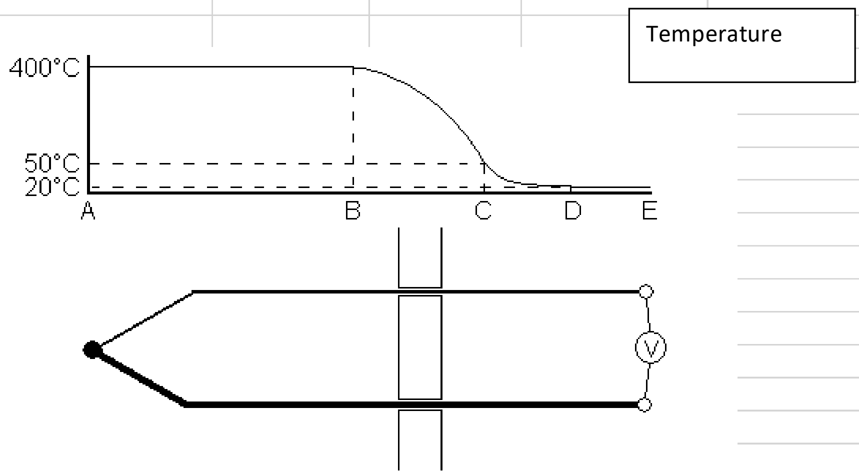


Figure 1. Schematic diagram showing the temperature gradient and immersion of thermocouple.

Please provide the length of A to B and A to C, A is the position of the tip of the thermocouple, B and C are the positions where temperature of the furnace, T drops to 90% and 10%.

B-8. NPLIA

Appendix B: Measuring equipment used in the comparison.

Laboratory Name: __________ NPLI India

Length of thermocouple wires:	i) As received	1380 mm
	ii) After measurement	1370 mm
	iii) Number of hours >1000°C	70 h

Devices	Type	Manufacturer	Serial number	Description	position of temperature gradient zone (mm)	
					A-B (tip to 90% T)	A-C (tip to 10% T)
Ice-Point used		NPLI	IPC-03	Stability 3 mK		
DVM used	8 &1/2	Fluke	8508A, Sr.No. 170062540	0.01 µV		
Scanner (if used)	NO					
Fixed Point /Enclosure used	Cu point	Isotech, UK	ISOTECH-F37405, Cu022	99.99995% Purity	34 cm	47 cm
Fixed points /Enclosure used	Co-C point	NPLI	Co-C-01	99.998% Purity	25 cm	39 cm
Fixed points / Enclosure used	Pd point	NPLI	Wire-bridge	Pd wire, 99.999% purity	32 cm	45 cm
Other enclosure	Metrology Furnace	Isotech, UK	Isotech Model 465, Sr. No. 221120-1	For Cu fixed point		
Other enclosure	Three-zone verticle Tube Furnace	Carbolite UK	TZF-16/610, Sr. No. 21-100894	For Co-C fixed point		
Other enclosure	Three-zone Horizontal Tube Furnace	Gero-Carbolite UK	TZF-16/610, Sr. No. 21-700648	For Pd fixed point with Alumina block, 30 cm		

APPENDIX C: Raw Data supplied by the Participants

C-1) KRISS, South Korea

Calibration Data of the Thermocouple

Serial Number: 2017-10 Inhomogeneity (From NMIA):

Name of Laboratory: **KRISS**

Initial

Temperature (Nominal T)	T_{ref} /°C	E_{ref} /µV	E_{meas} /µV	E_{meas} - E_{ref}/µV	Average E-E_{ref} /µV	Std Dev /µV	Uncertianty /µV (*k*=2)
1084.62 °C (Cu FP)	1084.62	11640.43	11639.6	-0.83	-1.36	0.55	1.18
			11639.1	-1.33			
			11638.5	-1.93			
1324 °C (Co-C MP)	1324.1	14968.3	14961	-7.3	-7.5	0.20	11.3
			14960.6	-7.7			
			14960.8	-7.5			
1554.8 °C (Pd FP)	1559.1	18279.3	18266.6	-12.7	-12.1	0.65	16.8
			18267.9	-11.4			
			18267.2	-12.1			

Final

Calibration Data of the Thermocouple (Second round)

Serial Number: 2017-10 Inhomogeneity (From NMIA):

Name of Laboratory: **KRISS**

Temperature (Nominal T)	T_{ref} /°C	E_{ref} /µV	E_{meas} /µV	E_{meas} - E_{ref}/µV	Average E-E_{ref} /µV	Std Dev /µV	Uncertianty /µV (*k*=2)
1084.62 °C (Cu FP)	1084.62	11640.43	11638.9	-1.5	-1.5	0.05	0.43
			11639.0	-1.5			
			11638.9	-1.6			
1324 °C (Co-C MP)	1324.1	14968.3	14960.3	-8.0	-8.2	0.18	11.3
			14960.1	-8.2			
			14959.9	-8.4			
1554.8 °C (Pd FP)	1559.1	18279.3	18270.8	-8.5	-8.6	0.45	16.8
			18270.2	-9.1			
			18271.0	-8.3			

C-2 NIM, CHINA

Appendix C: Calibration Data of the thermocouple

Serial Number: 2017-05 **Inhomogeniety (From NMIA)** ___________

Name of Laboratory: National Institute of Metrology

Initial

Temperature (nominal T)	**T_{ref} /°C** eg. after corrections or cell calibration	**E_{ref}/ µV** calculated from T_{ref}	**E_{meas}** /µV	**E_{meas} - E_{ref}** /µV	**Average** $E-E_{ref}$/µV	**Uncertainty (*k*=2)** /µV
1084 °C (eg. Cu point)	1084.62	11640.4	i) 11639.6 ii) 11639.3 iii) 1639.4	I) -0.8 ii) -1.1 iii) -1.0	-1.0	3.0
1324 °C (eg. Co-C point)	1234.0	14966.9	i) 14965.5 ii) 14965.7 iii)14965.9	i) -1.4 ii) -1.2 iii) -1.0	-1.2	5.5
1554°C (eg. Pd point)	1554.8	18219.2	i) 18213.3 ii) 18215.0 iii) 18214.0	i) -5.9 ii) -4.2 iii) -5.2	-5.1	8.2

Final

Serial Number: 2017-05 **Inhomogeniety (From NMIA)**

Temperature (nominal T)	**T_{ref} /°C** eg. after corrections or cell calibration	**E_{ref}/ µV** calculated from T_{ref}	**E_{meas}** /µV	**E_{meas} - E_{ref}** /µV	**Average** $E-E_{ref}$/µV	**Uncertainty (*k*=2)** /µV
1084 °C (eg. Cu point)	1084.62	11640.4	i) 11639.6 ii) 11639.5 iii)11639.9	I) -0.8 ii) -0.9 iii) -0.5	-0.73	3.0
1324 °C) (eg. Co-C point)	1234.0	14966.9	i) 14964.4 ii) 14964.3 iii) 4964.5	i) -2.5 ii) -2.6 iii) -2.4	-2.50	5.5
1554 °C (eg. Pd (point)	1554.8	18219.2	i) 18210.9 ii) 18210.6 iii)18212.1	i) -8.3 ii) -8.6 iii) -7.1	-8.00	8.2

C-3. NIMT, Thailand

Appendix C: Calibration Data of the Thermocouple

Serial Number _2017-07________Inhomogeneity (from NMIA)__-

____

Name of the Laboratory:___NIMT_________________________________

___

Initial

Temperature (nominal T)	**T_{ref} /°C** eg. after corrections or cell calibration	**E_{ref}/ µV** calculated from T_{ref}	**E_{meas}** /µV	**E_{meas} - E_{ref}** /µV	**Average** E-E_{ref}/µV	**Uncertainty*** (*k*=2) /µV
1084 °C (eg. Cu point)	1084.62	11640.4	i) 11639.6 ii) 11639.8 iii) 11639.9	i) -0.8 ii) -0.6 iii) -0.5	-0.6	1.2
1324 °C) (eg. Co-C point)	1324.06	14967.8	i) 14960.3 ii) 14960.0 iii) 14960.3	i) -7.5 ii) -7.8 iii) -7.5	-7.6	6.5
1554 °C (eg. Pd (point)	1553.5	18201.0	i) 18196.7 ii) 18196.8 iii) 18196.4	i) -4.3 ii) -4.2 iii) -4.6	-4.4	12.0

* Not included inhomogeneity value .

Serial Number _2017-07________Inhomogeneity (from NMIA)_

Final

Temperature (nominal T)	**T_{ref} /°C** eg. after corrections or cell calibration	**E_{ref}/ µV** calculated from T_{ref}	**E_{meas}** /µV	**E_{meas} - E_{ref}** /µV	**Average** E-E_{ref}/µV	**Uncertainty*** (*k*=2) /µV
1084 °C (eg. Cu point)	1084.62	11640.4	i) 11639.4 ii) 11639.6 iii) 11639.7	i) -1.0 ii) -0.8 iii) -0.7	-0.8	1.2
1324 °C) (eg. Co-C point)	1324.06	14967.8	i) 14960.0 ii) 14960.1 iii) 14960.3	i) -7.8 ii) -7.7 iii) -7.5	-7.6	6.5
1554 °C (eg. Pd (point)	1553.5	18201.0	i) 18195.0 ii) 18194.4 iii) 18194.5	i) -6.0 ii) -6.6 iii) -6.5	-6.3	12.0

C- 4) NMC, A*STAR, Singapore

Appendix C: Calibration Data of the thermocouple

Serial Number : 2017-06 Inhomogeneity (from NMIA)___

Name of the Laboratory: Temperature and Humidity Laboratory, National Metrology Centre, A*STAR

Initial

Temperature (nominal T)	T_{ref} /°C eg. after corrections or cell calibration	E_{ref}/ µV calculated from T_{ref}	E_{meas} /µV	$E_{meas} - E_{ref}$ /µV	**Average** $E-E_{ref}$/µV	**Uncertainty** (*k*=2) /µV
1084 °C (eg. Cu point)	1083.476 1083.019 1083.125	11624.903 11618.701 11620.140	11626.546 11621.711 11622.657	1.643 3.010 2.517	2.390	6.0
1324 °C) (eg. Co-C point)	1322.102 1322.241	14940.148 14942.108	14940.103 14942.696	-0.045 0.588	0.271	7.9
1554 °C (eg. Pd (point)	1553.557 1553.934	18201.799 18207.069	18204.105 18207.703	2.306 0.634	1.470	8.5

Serial Number : 2017-06 Inhomogeneity (from NMIA)_______________

Final

Method	**Temperature** (nominal T)	T_{ref} /°C eg. after corrections or cell calibration	E_{ref}/ µV calculated from T_{ref}	E_{meas} /µV	$E_{meas} - E_{ref}$ /µV	**Average** $E-E_{ref}$/µV	**Uncertainty** (*k*=2) /µV
Fixed Point	1084 Cu Fixed point	1084.580 1084.580 1084.580	11639.887 11639.887 11639.887	11638.684 11638.463 11638.679	-1.203 -1.424 -1.208	-1.279	1.7
Comparison	1084 °C (eg. Cu point)	1082.863 1083.086 1082.989	11616.578 11619.610 11618.294	11616.780 11620.290 11619.940	0.202 0.680 1.646	0.843	6.0
Comparison	1324 °C) (eg. Co-C point)	1321.076 1321.013 1321.064	14925.682 14924.793 14925.512	14923.790 14922.960 14923.670	-1.892 -1.833 -1.842	-1.856	7.9
Comparison	1554 °C (eg. Pd (point)	1550.748 1553.207 1553.074	18162.518 18196.905 18195.045	18165.030 18198.490 18196.910	2.512 1.585 1.865	1.987	8.5

C-5) NMIJ/AIST, JAPAN

Calibration Data of the Thermocouple

Serial Number:___2017-09___________ Inhomogeneity (from NMIA):_______________

Name of the Laboratory: ___NMIJ___

Initial

Temperature (nominal T)	**T_{ref}/°C** eg. after corrections or cell calibration	**E_{ref}/ µV** calculated from T_{ref}	**E_{meas} /µV**	**E_{meas} - E_{ref} /µV**	**Average $E-E_{ref}$/µV**	**Uncertainty (k=2) /µV**
1084 °C (eg. Cu point)	1084.62	11640.4	i) --- ii) --- iii) ---	i) --- ii) --- iii) ---	---	---
1324 °C (eg. Co-C point)	1324.0	14966.9	i) 14961.3 ii) 14961.2 iii) 14961.1	i) -5.6 ii) -5.7 iii) -5.8	-5.7	7.4
1554 °C (eg. Pd point)	1553.5	18201.0	i) 18188.5 ii) 18188.8 iii) 18188.9	i) -12.5 ii) -12.2 iii) -12.1	-12.3	6.5

Calibration Data of the Thermocouple

Serial Number:___2017-09___________ Inhomogeneity (from NMIA):_______________

Final

Temperature (nominal T)	**T_{ref}/°C** eg. after corrections or cell calibration	**E_{ref}/ µV** calculated from T_{ref}	**E_{meas} /µV**	**E_{meas} - E_{ref} /µV**	**Average $E-E_{ref}$/µV**	**Uncertainty (k=2) /µV**
1084 °C (eg. Cu point)	1084.62	11640.4	i) --- ii) --- iii) ---	i) --- ii) --- iii) ---	---	---
1324 °C (eg. Co-C point)	1324.0	14966.9	i) 14961.5 ii) 14961.2 iii) 14961.1	i) -5.4 ii) -5.7 iii) -5.8	-5.6	7.4
1554 °C (eg. Pd point)	1553.5	18201.0	i) 18189.0 ii) 18188.8 iii) 18189.4	i) -12.0 ii) -12.2 iii) -11.6	-11.9	6.5

C - 6) NMISA, South Africa

Appendix C: Calibration Data of the Thermocouple

Serial Number 2017-11 **Inhomogeneity (from NMIA)** _______________

Name of the Laboratory: National Metrology Institute of South Africa (NMISA)

Initial

Temperature (nominal T)	**T_{ref} /°C** eg. after corrections or cell calibration	**E_{ref}/ µV** calculated from Tref	**E_{meas}** /µV	**E_{meas} - E_{ref} /µV**	**Average** E-Eref/mV	**Uncertainty (k=2) /mV**
1084 °C (eg. Cu point)	1084.62	11640.43	i) 11637.943 ii) 11637.056 iii) 11636.402	i) -2.487 ii) -3.374 iii) -4.028	-3.296	1.87
1554 °C (eg. Pd point)	1553.5	18201.002	i) 18194.301 ii) 18194.865 iii) 18186.562	i) -6.701 ii) -6.137 iii)-14.439	-9.092	12.68

Name of the Laboratory: National Metrology Institute of South Africa (NMISA)

Final

Temperature (nominal T)	**T_{ref} /°C** eg. after corrections or cell calibration	**E_{ref}/ µV** calculated from Tref	**E_{meas}** /µV	**E_{meas} - E_{ref} /µV**	**Average** E-Eref/mV	**Uncertainty (k=2) /µV**
1084 °C (eg. Cu point)	1084.62	11640.43	i) 11637.817 ii) 11636.883 iii) 11636.736	i) -2.613 ii) -3.547 iii) -3.694	-3.285	1.519
1554 °C (eg. Pd point)	1553.5	18201.002	i) 18200.146 ii) 18199.901 iii) 18186.099	i) -0.857 ii) -1.101 iii)-14.902	-5.62	12.2996

C - 7) BSN, Indonesia

Appendix C: Calibration Data of the Thermocouple

Serial Number : 2017-08 **Inhomogeneity (from NMIA)**_______________

Name of the Laboratory: RCM-LIPI

Initial

Temperature (Nominal *t*)	t_{ref} (°C)	E_{ref} (μV)	E_{meas} (μV)	E_{meas} - E_{ref} (μV)	**Average** (E_{meas} - E_{ref}) (μV)	**Uncertainty** (k=2) (μV)
1084 °C (eg. Cu point)	1084	11632.0292	11622.17728	-9.8519	-7.8	11
		11632.0144	11623.62422	-8.3902		
		11632.0141	11626.74264	-5.2715		
1324 °C (eg. Co-C point)	1324	14966.9142	14954.31181	-12.6024	-11	15
		14966.9125	14956.91353	-9.9990		
		14966.9126	14956.67149	-10.2411		
1554 °C (eg. Pd point)	1554	18207.9919	18183.527	-24.4649	-28	32
		18207.9926	18181.40918	-26.5835		
		18207.9924	18175.01583	-32.9765		

Serial Number : 2017-08 **Inhomogeneity (from NMIA)**_______________

Final

Temperature (Nominal *t*)	t_{ref} (°C)	E_{ref} (μV)	E_{meas} (μV)	E_{meas} - E_{ref} (μV)	**Average** (E_{meas} - E_{ref}) (μV)	**Uncertainty** (k=2) (μV)
1084 °C (eg. Cu point)	1084	11632.0144	11624.77972	-7.2347	-6.2	17
			11626.32425	-5.6902		
			11626.32673	-5.6877		
1324 °C (eg. Co-C point)	1324	14966.9125	14955.32657	-11.5859	-12	22
			14955.7473	-11.1652		
			14954.89153	-12.0210		
1554 °C (eg. Pd point)	1554					

C - 8) NPLI, India

Serial Number : 2017-02 **Inhomogeneity (From NMIA) :**

Name of the Laboratory : NPLI India **Initial**

Temperature (nominal T)	T_{ref} /°C eg. after corrections or cell calibration	E_{ref}/ µV calculated from T_{ref}	E_{meas} /µV	E_{meas} - E_{ref} /µV	Average $E\text{-}E_{ref}$/µV	Uncertainty (k=2) /µV
1084 °C (eg. Cu point)	1084.62	11640.68	i) 11641.27 ii) 11640.67 iii) 11640.32	i) 0.59 ii) -0.01 iii) -0.36	0.07	3.29
1324 °C) (eg. Co-C point)	1323.93	14966.02	i) 14967.85 ii) 14967.08 iii) 14966.96	i) 1.83 ii) 1.06 iii)0.94	1.28	5.13
1554 °C (eg. Pd (point)	1554.8	18219.2	i) 18217.23 ii)18216.90 iii)18218.14	i) -1.97 ii) -2.30 iii) -1.06	-1.78	6.07

Final

Serial Number : 2017-02 **Inhomogeneity (From NMIA) :**

Name of the Laboratory : NPLI India

Temperature (nominal T)	T_{ref} /°C eg. after corrections or cell calibration	E_{ref}/ µV calculated from T_{ref}	E_{meas} /µV	E_{meas} - E_{ref} /µV	Average $E\text{-}E_{ref}$/µV	Uncertainty (k=2) /µV
1084 °C (eg. Cu point)	1084.62	11640.68	i) 11636.89 ii) 11636.38 iii) 11636.63	i) -3.79 ii) -4.30 iii) -4.05	-4.04	3.25
1324 °C) (eg. Co-C point)	1323.93	14966.02	i) 14960.06 ii) 14961.03 iii) 14960.49	i) -5.96 ii) -4.99 iii)-5.53	-5.49	5.14
1554 °C (eg. Pd (point)	1554.80	18219.20	i) 18212.43 ii) 18214.03 iii) 18213.36	i) -6.77 ii) -5.17 iii) -5.84	-5.93	6.09

APPENDIX D: Calibration Procedure used by the participants

D -1) Calibration procedure of KRISS

1. Realization of fixed-points.
a) Ice point: Finely crushed ices were mixed with purified water in the dewar flask. The dewar has a maximum immersion depth of 285 mm. Each t/c legs were connected with pure Cu wires having diameter of 0.5 mm and the junctions were immersed into small diameter pyrex tubes.
b) Cu freezing point: Open type Cu cell made by KRISS was installed into 3-zone vertical electric furnace. High purity Ar more than 99.999 % containing 0.5 vol% of hydrogen gas was introduced into the cell during realization. Cu was melted at about 1090 ºC and cooled down to 1081 ºC allowing supercool and recovery. The tip position was controlled to about 5 mm above the cell bottom. To check the conduction error, t/c was slightly moved up by 1 cm during freezing.
c) Co-C melting point: Open type Co-C cell made by KRISS, which was the same as the APMP.T-S7, was used. Carrying gas was same to the case of Cu. The cell temperature was determined using type R thermocouple, which was calibrated with a reference grade radiation thermometer, LP4, at the horizontal comparison furnace. LP4 was calibrated at the Cu freezing point in KRISS. The tip position was controlled to about ~2 mm above the cell bottom. Realization processes were exactly same as the case of APMP.T-S7. The melting emf was determined from the inflection point of the melting plateau. To check the conduction error, t/c was moved up by 1 cm and another melting was performed.
d) Pd freezing point: Open type Pd cell was made by KRISS using an alumina crucible with length of 100 mm. It contained about 100 g of pure Pd with purity of 99.995 % from Alfa Aesar. Carrying gas was same to the case of Cu. The cell temperature was determined using type B thermocouple, which was calibrated with a reference grade radiation thermometer, LP4. The freezing emf was determined from the maximum point of the freezing plateau. To check the conduction error, t/c was moved up by 1 cm and another melting was performed.
2. Data measurement
DVM was turn on for the stabilization before test few days ago. The display was set to zero using 'REL' function of the DVM. All data were measured through a laptop PC with 3 second interval.

D – 2) Calibration Procedures – NIM, China

Inspection

The wooden box was sent to our laboratory by on 4th Aug. 2017. It was opened for careful inspection. The APMP .T-S16 transfer thermocouple (serial number 2017-05) was ok.

Preparation

The reference junction of thermocouple should to assembled by myself according to the protocol. The length of both legs of thermocouple was measured and recorded. A pair of twist copper wires with ø 0.5mm and 1.5 m length was welded to the ends of that reference junction of thermocouple, where it was fixed and insulated with heat-shrink PVC tube, and then inserted into the bottom of an end closed stainless steel tube (210mm long and 6mm OD) and sealed. The reference junction of thermocouple was inserted 20cm deep into a bottle of ice point which was made of crash ice and water mixture in Dewar vessel in whole measurement.

The thermocouple was inserted into an alumina protection tube and fixed its tip about 1.5cm above the bottom of protection tube, which was Al_2O_3 99.7%, an end-closed, 7.6mm OD, 4.5mm ID and 710mm long, and also baked under 1100°C for 3h before being used during Cu FP and Co-C HTFP calibration .

The electromotive voltages of thermocouple was measured by a 2182 Nanovoltmeter from Keithley and a 160B scanner from Data Proof, which were applied to automatic data acquisition at intervals of 2s during the whole measurement period. The voltage was traced to NIM.

Cu FP calibration

The copper cell was a sealed structure, and a Na heat pipe was installed in the fixed point furnace, which was manufactured by Hart Scientific. The temperature of cell was traced to the NIM reference group.

The thermocouple assembly was inserted into the bottom of the cell in freezing plateau. The temperature of furnace was kept at 0.5℃ lower than freezing point temperature. When the thermocouple was in thermal equilibrium with the cell, the electromotive voltages of thermocouple was continuously recorded for 30min at freezing plateau.

Co-C HTFP calibration

The home-made Co-C #1 cell assembly and Mat-60SC2 high temperature furnace (from Chino) was used in Co-C HTFP calibration. The ITS-90 temperature of Co-C cell was assigned by radiation thermometer (LP4 model). The measurement of crucible temperature was made, at the original position where the thermocouple calibration was done, through a mirror which reflected the light of the vertically installed crucible to the horizontally placed radiation thermometer. The radiation thermometer was calibrated by Cu FP before and after measurement to remove drift. The temperature of radiation thermometer was traceable to NIM standard.

A high temperature furnace prepared for calibration was tuned at 1320℃, the temperature uniformity around the crucible was better than ±0.5℃. The crucible is 41mm in diameter and 120mm in length. The thermocouple assembly was installed in the furnace and inserted into the bottom of the crucible before measurement. The enclosure was sealed, evacuated, and flushed with Argon 3 times before the furnace was heated up. The temperature of furnace was raised to 1314°Cand kept for 1h for stability, then the furnace temperature was raised to 1334°C for +10K, held at that point for 1h for fully melting and then dropped to 1314°C for -10K, held at that point for 1h for freezing completely. This process was repeated for 3 times. The thermocouple was left 1~2cm to estimate the effect brought by heat flux.

Palladium MP calibration

The protection tube was removed from thermocouple before measurement. A Palladium mini coil was fixed in the measurement junction of thermocouple. The Palladium wire from Alfa Aesar had a nominal purity of 99.99+% and a diameter of 0.5mm. The Pd wire cleaned with ethanol was winded onto a 0.6mm OD plastic rod in 4 rings then cut off. A end-closed alumina tube was inserted into a vertical high temperature furnace at the depth of 500mm, and the 99.999% Argon was injected by a slim alumina tube into it with a flow rate at 100mL/min to keep the air out. The furnace temperature was set to 1550℃ and kept invariable for 30 min, then thermocouple was inserted into the working zone, holding for 20 min for thermal equilibrium. The furnace temperature was controlled at a ramp rate of 0.5℃/min from 1550℃ to 1560℃. When Palladium melting completed, the thermocouple was pulled out quickly. The measurement junction of thermocouple was cut by 5mm, and a new Palladium Mini Coil was installed and oxyhydrogen flame was used to re-weld the junction of thermocouple and the mini coil was secured. Pd wire-bridge calibration was repeated for 3 times, in last of which the measurement junction of thermocouple was cut off again and the lengths of positive and negative elements of thermocouple were measured and recorded.

• D – 3) Calibration Procedure _NIMT

Name of laboratory : NIMT-Thailand

The thermocouple type R S/N: 2017-07 was received from NMIA under APMP Regional comparison. The artifact has been checked for any physical damage to the instrument. The pair of Cu wires was connected to TC wires to be the cold junction. Thermocouple was calibrated using fixed points of Cu, Co-C and the melting point of Pd wire. The reference cold junction is an ice point. The emf of thermocouple measured by using DVM which is traceable to NIMT. The measurement is performed from lower to higher.

Calibration at Cu fixed point cell: The melting and freezing plateau was prepared using the reference thermocouple. After the freezing plateau is reached for 1 hr, then the reference thermocouple is removed from the cell and the preheated artifact is measured. After finishing the calibration, the artifact is withdrawn from the cell to room temperature slowly. The emf result is the average of 20 data at the plateau. The Cu fixed point was realized three times.

Calibration at Co-C eutectic fixed point cell: the thermocouple was inserted into the Co-C apparatus at the room temperature. Then the furnace temperature was raised up (rate 5 °C/min) to temperature 10 °C below the melting temperature and holding for 1 hr. After that the furnace controller is increased (2 °C/min) to temperature 10 °C above the melting temperature. After the melting finished, the furnace controller is decreased (2 °C/ min) to temperature 10 °C below the melting/freezing temperature. During all measurement, the gentle flow of argon is used. The melting plateau of Co-C cell was realized three times within 6 hr measurement. The emf result was calculated from the inflection point of the melting plateau.

Pd melting wire: Attaching the 5-6 coils shaped 0.5 mm Pd wire to the measurement junction of thermocouple. Set the furnace controller to approximately 950 °C. At this temperature, gently immerse UUC with Pd coils into the most uniform and highest temperature of the furnace. Then, set the furnace controller temperature rate of 2.5 °C/min to approx. 5 °C below the melting temperature (approximate 1549 for Pd wire bridge). Allow the system to be stable for 1 hr then set the furnace controller to approx. 3 °C above the melting point temperature (Scan rate 1 °C / min). After melting finish, then cool down the furnace. The thermocouple was gently removed from the furnace at about 950 °C. The thermocouple tip with melted Pd was cut. Repeat the process three times. The emf result was obtained from the inflection point of the Pd melting plateau.

D – 4) General outline of the calibration procedure _NMC, A*STAR

1) Inspection and Preparation for the Comparison

The artefact (Type R thermocouple) was sent to NMC, A*STAR by A Sonic Logistics on 5 July 2017. The packaging was opened carefully and the thermocouple was inspected for damage. It was found that there was a small defect at the corner of the ceramic sheath near the measuring junction of the thermocouple and this was reported to the pilot (NMIA) immediately. The pilot lab commented that it should not make any impact in the measurement. The rest conditions of the thermocouple were normal. We measured the total length of the thermocouple wire (1380 mm) and the length of the bi-core sheath (720 mm). A pair of Cu-wires to the CJ (cold-junction) of the thermocouple was prepared and assembled to the thermocouple.

A blackbody furnace, specially designed for thermocouple calibration, and a radiation thermometer [1] were used as transfer temperature source and temperature reference, respectively, for all the measurement points. The furnace consists of three independent heating zones with heating elements made of molibdenum disilicite (MoSi2) 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. The thermocouple under measurement was positioned just behind the back wall of the blackbody cavity with an immersion depth about 400 mm. The radiation thermometer works at a fixed focusing distance of 505 mm with a target size of 1.0 mm at 0.65 µm. It uses a silicon photodiode made by Hamamatsu model S2386-5K. The working wavelength is defined by a hard-coated interference filter with bandpass of 10 nm. A PT-100 is installed near the filter carrier for compensation of the temperature coefficient of the thermometer. The T_{90} carried out by the radiation thermometer is established according to ITS-90 definition. Copper fixed point and silver fixed point were realized by freezing plateau every day to be taken as the reference values for the radiation thermometer. The signal output of the radiation thermometer was measured by a multimeter. A data acquisition software was used for data acquisition from the multimeter.

In order to minimize the error due to a possible axial temperature gradient of the furnace, two type B thermocouples were used to evaluate the axial temperature uniformity. During the evaluation, the main and slave controllers of the furnace were adjusted so that the temperature uniformity was within ± 0.15 ºC at an 100 mm working zone for all the test points.

In the preparation stage, an alumina sheath was baked at 950 ºC for 30 minutes, inserting the artefact into the sheath to protect it from contamination while using the furnace.

2) Calibration Procedure

When the thermocouple was in thermal equilibrium with the furnace, the electromotive voltages (emf) of thermocouple was measured using a Keithley 2182 Nanovoltmeter together with a scanner (see attached file **Instrument.doc** for details of the equipment) and recorded for 30 mins at each melting plateau.

From lowest to highest temperature points, measurements were accomplished by using the furnace and the radiation thermometer for 2 or 3 times at each temperature point. When the furnace was stabilized at each test point, the measurement for both artefact and radiation thermometer were taken simultaneously for at least 30 readings (3 minutes). The average reading for the 30 readings for both artefact and radiation thermometer were taken as the final result.

In order to cross check the measurement result using the furnace at Cu fixed point temperature, we conduct the measurement using our mini Cu fixed point cell. For the Cu fixed point cell measurement, the full immersion length of the artefact was 20 mm above the cell bottom. The measurement was completed by using artefact without alumina sheath. The average reading of 3 realizations was taken as the final result. The agreement between the two methods is within 1.47 µV.

[1] M. Battuello, F. Girard, L. Wang, *INRIM-NMC Comparison of Pt/Pd Calibration Above the Ag Point*, International Journal of Thermophysics (2010) 31: 1444-1455

D – 5) NMIJ

General outline of the calibration procedure

1) A pair of Cu wires were connected to the cold-junction ends (CJ ends) of the transfer-thermocouple wires.
2) The measurement at Cu fixed point was skipped.
3) The transfer thermocouple was calibrated at Co-C point using a Co-C cell. The melting temperature of the Co-C cell was determined using a calibrated radiation thermometer.
4) Before the calibration of the transfer thermocouple at the Co-C point, the conduction error for the Co-C cell was investigated during the melting by moving a monitoring thermocouple by 2 cm pitch upward and downward alternately along the thermometer well.
5) For the calibration at the Co-C point, the transfer thermocouple was inserted 500 mm below the top of the Co-C point furnace used. During three pairs of melting and freezing plateaux in the Co-C cell, the CJ ends of the transfer thermocouple were immersed 230 mm into a mixture of shaved ice and distilled water in a Dewar flask.
6) After measurements at the Co-C point, the transfer thermocouple was inserted 700 mm into a horizontal annealing furnace, then annealed at 1100 °C for 45 minutes.
7) After annealing at 1100 °C, the transfer thermocouple was calibrated at Pd melting point by Pd melt-wire technique. The nominal purity of Pd wire was 99.99%.
8) For performing Pd melt-wire technique, a coil-shaped Pd wire was attached to a hot-junction of the transfer-thermocouple wires. The transfer thermocouple was inserted 380 mm below the top of the Pd point furnace used. After melting the attached Pd wire in air, the transfer thermocouple was withdrawn from the furnace, and then its wires were cut approximately 5 mm from the hot-junction with the melted Pd wire. During the melting of the attached Pd wire, the CJ ends of the thermocouple was immersed 230 mm into a mixture of shaved ice and distilled water in a Dewar flask. The Pd melting point was realized three times.
9) After the measurements at Pd melting point, the Cu wires were removed from the CJ ends of the transfer-

thermocouple wires.

D – 6) Laboratory name: **National Metrology Institute of South Africa (NMISA)**

Thermocouple calibration at fixed points is conducted based on the relevant procedures at NMISA. In measuring the thermocouple at Cu fixed point the following procedure was followed:

- Prepare the thermocouple by cleaning it with absolute ethanol
- Prepare ice for the thermocouple reference junction.
- Pre-heat the thermocouple at Cu point temperature
- Argon with purity level of 99.999 % (as an open Cu cell is used) is used
- Prepare the Cu cell (in Argon environment) for fixed point realization by adjusting the set point of the furnace 2 - 5 °C above the Cu point. When the ingot starts to melt the thermocouple reading will stabilize. Stable reading is recorded
- After the ingot is completely melted it is allowed to soak at temperature 2-5 °C above fixed point temperature over night.
- Furnace controller offset is determined by using the ratio of temperatures measured at 2-5 °C above and below melt point.
- Furnace set point is changed to 2-5 °C below Cu fixed point considering the newly determined furnace offset.
- When recalescence occurs, the furnace set point is reduced to 1 °C below fixed point temperature.
- A room temperature kept alumina or quartz rod was inserted into the re-entry well 2-3 times to initiate the freeze after removing and putting the thermocouple in a pre-heat well.
- The thermocouple that was kept at a pre-heat well was inserted in the re-entry well and when stable the reading was recorded.
- On a freeze plateau uncertainty due to conduction was determined by incrementally raising the thermocouple.
- The melt/freeze was repeated three times.
- The fixed point temperature was determined from a freeze plateau. The average of the three freezes was considered as a fixed point temperature.

In measuring at Pd point in air using wire bridge method the following procedure was followed:

- Melt a globule on one end of the Palladium (Pd) wire and fuse it to one leg of the thermocouple. Cut off about 8mm of Pd wire and melt globule on the loose end. Bend it carefully with clean long-nose pliers, and fuse it to the other leg of the thermocouple.
- Reconstruct the reference junction and put it in ice.
- Put the measuring junction in the middle depth of the furnace.
- Increase the temperature of the furnace slowly.
- At the moment when the bridging wire begins to melt the emf of the thermocouple becomes static for a moment or two. After the melt is complete the Pd may fall off causing the thermocouple to go open circuit. If the Pd does not fall off the emf of the thermocouple will again start increasing as the measuring junction warms up to the furnace temperature.
- The static value of the emf of the thermocouple is taken as its output at Palladium point
- Decrease the temperature of the furnace so that the thermocouple can cool down slowly
- After the thermocouple has cooled down below 1100 °C the thermocouple may be removed and reconstructed.

D – 7) Calibration Procedure: Name of Laboratory: NPLI – INDIA

1. The Type-R Thermocouple (2017-02) was received at CSIR-National Physical Laboratory (NPLI), New Delhi 110 012, India on 09/08/2017. The thermocouple was in good physical and working condition. The Cold Junction and copper connecting wires were prepared for the measurements.

2. According to the protocol, we have used the fixed points of FP of Cu (1084.62 °C), MP of Co-C (1323.93 °C) and MP Pd (1554.8 °C, by wire-bridge method) for the inter-comparison measurements.

3. First, we have used the Metrology Furnace (Isotech Model 465) and the temperature profile was measured at 1074 °C. The fixed point cell of Copper (sealed cell, Isotech UK, Model –ISOTECH –F37405-Cu022, purity 99.99995%) was used to measure the melting-freezing plateau's for 3 times. The uncertainty of the FP of Cu temperature was calculated by taking the components suggested in the protocol such as, purity, melt-freeze agreement, choice of fixed point and other the realization components of fixed point.

4. For the realization of MP of Co-C the Three –zone vertical tube furnace (Carbolite UK, Model TZF 16/610, 1600 °C) was used. NPLI developed Co-C cell (Co-C-01) was used for the measurement of three melting-freezing plateau's. The temperature was assigned to Co-C melting point (1393.93 °C) using radiation thermometry and the corresponding uncertainties are used.

5. For the realization of MP of Pd by wire bridge method the Three –zone horizontal tube furnace (Gero-Carbolite UK, Model TZF 16/610, 1600 °C) with Alumina thermalizing Block was used. Three melting points of Pd were recorded. The uncertainty of

temperature realization at MP of Pd (1554.8 ^{o}C) was evaluated using the reference thermocouple.

6. For the evaluation of thermocouple stability during calibration, the FP of Cu was measured after the MP of Pd realizations, and corresponding uncertainty was taken in the evaluation of Co-C and Pd point.

7. For the $E-E_{REF}$ at the corresponding fixed point temperatures, NIST ITS-90 thermocouple database was used.

D – 8) BSN - Indonesia Procedure for Calibration of Type R Thermocouple

An artefact, type R thermocouple with serial number: 2017-08, was calibrated against two SI-traceable thermocouple standards: Pt/Pd and type B thermocouples on a single-zone-controller horizontal furnace heated by six robust SiC heating elements (Landcal P1600B, Land Instruments, Meerbusch, DE). The reference thermocouple EMFs at Cu, Co-C, and Pd points were obtained from the measurements using the standards. The Pt/Pd thermocouple (NPL 07/17/B, NPL, Teddington, UK) was used for the calibration at Cu and Co-C points, whilst the type B thermocouple (SIMP 61889, Tempsens Instruments, Udaipur, IN) was used for the calibration at Pd point because Pt/Pd thermocouple can no longer work at this temperature.

The artefact was allowed to equilibrate within a controlled, stable environment inside the furnace along with the standard. The position of standard must be as close as possible to the position of the artefact. Both thermocouples were places in the furnace at an immersion depth of about 370 mm. The reference junctions of Pt/Pd and Type R thermocouples were maintained at 0 °C. The readings of the thermocouples were recorded at each specified reference points. Reference EMFs was calculated by converting the reference thermocouple temperature with respect to the type of the thermocouple under calibration (type R thermocouple). The EMF-temperature equations given in the standard ASTM E-230 (1) and in the Ref. (2) were used for calculating the reference values of type B and Pt/Pd thermocouples, respectively. The calibration data, thermocouple-EMF error ($EMF_{meas}-EMF_{ref}$), were calculated by subtracting the measured EMF from the reference EMF. In order to obtain the values of thermocouple-EMF error exactly at the fixed points temperature (1084 °C, 1324 °C and 1554 °C), two measurements of thermocouple-EMF error close to the specified fixed-point temperatures were taken and a linear regression was employed to the results.

References:

1. Standard A. E230/E230M− 12. Standard Specification and Temperature-Electromotive Force (emf) Tables for Standardized Thermocouples, ASTM International, West Conshohocken, Pennsylvania. 2012.

2. Burns GW, Ripple DC, Battuello M. Platinum versus palladium thermocouples: an emf-temperature reference function for the range 0° C to 1500° C. Metrologia. 1998;35(5):761.

Appendix E: Uncertainty submitted by the Participating laboratories.

1) NMIA, Australia

Co-C point					
Source	**Uncertainty**	**ki**	**Description**	**ui**	**vi**
Temperature assigned on the cell	7.00	2.0		3.50	60
Quality of metal	0.50	2.0	normal	0.25	8
Determination of melt palteau	1.00	2.0	normal	0.50	20
Scatter/reproducibility in 3 melting points*	0.20		S.D. of data	0.12	4
DVM calibration	0.10			0.04	5
DVM drift in use	0.10	2.0	95% C.L..	0.05	60
Conduction error	0.10	2.0	95% C.L.	0.05	60
Measurment of CJ temperature	0.10	2.0	normal	0.05	20
Thermal and ac pickup	0.50	2.0	normal	0.25	20
Rounding error	0.10	1.7	rectangular	0.06	60
		u_c =		**3.56**	
Expanded Uncertainty U=	**7.11**	**k =**	**2.00**	**eff.v =**	**63.9**
	0.50	**°C**			

Pd-point					
Source	**Uncertainty**	**ki**	**Description**	**ui**	**vi**
Temperature on ITS-90	1.50		normal	0.75	20
Quality of metal	1.00	2.0	normal	0.50	20
Interpretation of melting plateau	1.00	2.0	normal	0.50	20
Scatter in 3 melting points	0.50		S.D. of data	0.29	2
DVM calibration	0.10	2.0	95% C.L.	0.05	20
DVm drift in use (4ppm/yr)	0.10	2.0	95% C.L.	0.05	20
Measurement of CJ temperature	0.10	2.0	normal	0.05	8
Rounding	0.10	1.7		0.06	60
Thermal and ac pickup	1.00	2.0	normal	0.50	8
		u_c =		**1.19**	
Expanded Uncertainty U=	**2.37**	**k =**	**2.00**	**eff.v =**	**59.3**
	0.17	**°C**			

Cu Point					
Source					
	Uncertainty	**ki**	**description**	**ui**	**vi**
Quality of metal	0.5	2	normal	0.25	20
Reproducibilty of freezing point (3 plateau)	0.50		*S.D. of data	0.29	2
Interpretion of flat	0.50	2.0	normal	0.25	20
Quality of metal	0.50	2.0	normal	0.25	20
DVM calibration	0.10	2.0	95% C.L..	0.05	60
DVM drift (4ppm/yr)	0.10	2.0	95% C.L.	0.05	60
Conduction error	0.10	2.0	normal	0.05	20
Measurment of CJ temperature	0.10	2.0	normal	0.05	20
Rounding	0.10	1.7	rectangular	0.06	60
Thermal and ac pickup	0.50	2.0	normal	0.25	20
	u_c =			**0.53**	
	k =		**2.10**	**eff.v =**	**19.0**
Expanded Uncertainty U=	**1.12**	0.08	°C		
		0.08	**°C**		

NMC, A*STAR

Calibration uncertainty of Type R thermocouple using radiation method

Reference RT

Type R

Sensitive coefficient of test TC at 0 °C (1iV/°C)	5.29
Sensitive coefficient of test TC at 1084.62 °C (1iV/°C)	13.575046
Reading at 1084.62°C (1iV)	11640.43

No.	Sources of uncertainty	Type	Uncertainty value	Probability Distribution	Coverage Factor	Standard uncertainty [u(Xi)]	Sensitivity coefficient [ci]	[Ici* u(xi)]	Degrees of freedom
1	Test temperature (°C)	B	0.190	normal	1	0.19000	13.575	2.5793	100000
2	TC indicator (1iV)	B	0.749	rectangular	1.732	0.43257	1.000	0.4326	100000
3	TC indicator resolution (µV)	B	0.005	rectangular	1.732	0.00289	1.000	0.0029	100000
4	TC extension leads (1iV)	B	0.000	rectangular	1.732	0.00000	1.000	0.0000	100000
5	Reference ice-point (°C)	B	0.002	rectangular	1.732	0.00115	13.575	0.0157	100000
6	TC inhomogeneity error (°C)	B	0.000	rectangular	1.732	0.00000	13.575	0.0000	100000
7	TC repeatability (°C)	A	0.006	t-distribution	1	0.00639	13.575	0.0867	29
8	TC reproducibility (µV)	B	1.500	rectangular	1.732	0.86605	1.000	0.8661	2
9	TC immersion error (°C)	B	0.000	rectangular	1.732	0.00000	13.575	0.0000	100000
10	Thermal EMF error (µV)	B	0.000	rectangular	1.732	0.00000	1.000	0.0000	100001
11	Multimeter Zeroing error (µV)	B	0.100	rectangular	1.732	0.05774	1.000	0.0577	100001
12	Data Proof Scanner (1iV)	B	0.020	rectangular	1.732	0.01155	1.000	0.0115	100000
13	Furnace Unifomrty(°C)	B	0.150	rectangular	1.732	0.08661	13.575	1.1757	100000

standar uncertainty	3.00	1iV	0.221 °C	
Effective degrees of freedom	286			
Coverage factor	2			
Expanded uncertainty (U)	5.99	1iV	0.442	°C
Roundup	6.0	1iV	0.45	°C

Calibration uncertainty of Type R thermocouple using radiation method

Reference RT

Type R

Sensitive coefficient of test TC at 0 °C (1iV/°C) 5.29

Sensitive coefficient of test TC at 1324 °C (1iV/°C) 14.102

Reading at 1324°C (1iV) 14966.91

No	Sources of uncertainty	Type	Uncertainty value	Probability Distribution	Coverage Factor	Standard uncertainty [u(Xi)]	Sensitivity coefficient [ci]	[Ici I* u(xi)]	Degrees of freedom
1	Test temperature (°C)	B	0.260	normal	1	0.26000	14.102	3.6665	100000
2	TC indicator (1iV)	B	0.849	rectangular	1.732	0.49019	1.000	0.4902	100000
3	TC indicator resolution (µV)	B	0.005	rectangular	1.732	0.00289	1.000	0.0029	100000
4	TC extension leads (1iV)	B	0.000	rectangular	1.732	0.00000	1.000	0.0000	100000
5	Reference ice-point (°C)	B	0.002	rectangular	1.732	0.00115	14.102	0.0163	100000
6	TC inhomogeneity error (°C)	B	0.000	rectangular	1.732	0.00000	14.102	0.0000	100000
7	TC repeatability (°C)	A	0.005	t-	1	0.00470	14.102	0.0663	29
8	TC reproducibility (µV)	B	0.060	rectangular	1.732	0.03464	1.000	0.0346	1
9	TC immersion error (°C)	B	0.000	rectangular	1.732	0.00000	14.102	0.0000	100000
#	Thermal EMF error (µV)	B	0.000	rectangular	1.732	0.00000	1.000	0.0000	100001
#	Multimeter Zeroing error (µV)	B	0.100	rectangular	1.732	0.05774	1.000	0.0577	100001
#	Data Proof Scanner (1iV)	B	0.020	rectangular	1.732	0.01155	1.000	0.0115	100000
#	Furnace Unifomrty(°C)	B	0.150	rectangular	1.732	0.08661	14.102	1.2213	100000

standar uncertainty	3.90 1iV		0.276 °C	
Effective degrees of freedom	#####			
Coverage factor	2			
Expanded uncertainty (U)	7.79	1iV	0.553	°C
Roundup	7.9	1iV	0.56	°C

Calibration uncertainty of Type R thermocouple using radiation method
Reference RT

Type R

Sensitive coefficient of test TC at 0 °C (µV/°C)	5.29
Sensitive coefficient of test TC at 1555 °C (µV/°C)	13.979
Reading at 1555°C (µV)	18221.97

No.	Sources of uncertainty	Type	Uncertainty value	Probability Distribution	Coverage Factor	Standard uncertainty [u(Xi)]	Sensitivity coefficient [ci]	[|ci|* u(xi)]	Degrees of freedom
1	Test temperature (°C)	B	0.280	normal	1	0.28000	13.979	3.9141	100000
2	TC indicator (µV)	B	0.947	rectangular	1.732	0.54657	1.000	0.5466	100000
3	TC indicator resolution (µV)	B	0.005	rectangular	1.732	0.00289	1.000	0.0029	100000
4	TC extension leads (µV)	B	0.000	rectangular	1.732	0.00000	1.000	0.0000	100000
5	Reference ice-point (°C)	B	0.002	rectangular	1.732	0.00115	13.979	0.0161	100000
6	TC inhomogeneity error (°C)	B	0.000	rectangular	1.732	0.00000	13.979	0.0000	100000
7	TC repeatability (°C)	A	0.006	t-	1	0.00641	13.979	0.0896	29
8	TC reproducibility (µV)	B	0.950	rectangular	1.732	0.54850	1.000	0.5485	1
9	TC immersion error (°C)	B	0.000	rectangular	1.732	0.00000	13.979	0.0000	100000
10	Thermal EMF error (µV)	B	0.000	rectangular	1.732	0.00000	1.000	0.0000	100001
11	Multimeter Zeroing error (µV)	B	0.100	rectangular	1.732	0.05774	1.000	0.0577	100001
12	Data Proof Scanner (µV)	B	0.020	rectangular	1.732	0.01155	1.000	0.0115	100000
13	Furnace Unifomrty(°C)	B	0.150	rectangular	1.732	0.08661	13.979	1.2107	100000

Combine standard uncertainty	4.17 µV		0.298 °C	
Effective degrees of freedom	3259			
Coverage factor	2			
Expanded uncertainty (U)	8.34	µV	0.597	°C
Roundup	8.5	µV	0.61	°C

NIM, China

Uncertainty Analysis of R Type thermocouple (2017-05) at Copper freezing Point

Unceratiny factors	Quantity	Probability Distribution	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution /uV	Remarks
fixed point temperature	0.2°C	Normal	0.1°C	13.57	1.36	tracibility to National temperature Reference of ITS-90
Measurement scatter	0.3j.iV	Normal	0.18j.iV	1	0.18	maximum difference of EMF in 3 time measurements
Conduction Errors	0.8j.iV	Rectangular	0.46j.iV	1	0.46	Emf variation between difference depth
Inhomogeneity						
CJ temperature	±0.02°C	Rectangular	0.012°C	5.3	0.06	impurity of water and conduction of stainless steel tube
DVM calibration	0.2j.iV	Normal	0.1j.iV	1	0.10	calibration uncertainty at 100 mV range
DVM Short-term stability	± 0.59j.iV	Rectangular	0.34j.iV	1	0.34	90 Day accuracy 25ppm*reading+3ppm*range
Stray EMF /Electric noise	± 0.1j.iV	Rectangular	0.06j.iV	1	0.06	AC pick up and electric leakage of heater
Rounding Error	± 0.1j.iV	Rectangular	0.06j.iV	1	0.06	Rounded to the nearest 0.1j.iV
Combined standard uncertainty /uV					1.49	

Expanded uncertainty (k = 2) /uV	2.99
Expanded uncertainty (k = 2) /ºC	0.22

NIM, China

Uncertainty Analysis of R Type thermocouple (2017-05) at Co-C HTFP

Unceratiny factors	Quantity	Probability Distribution	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution /j.iV	Remarks
Fixed point temperature	0.3°C	Normal	0.15°C	14.1	2.10	ITS-90 temperature assignment by Radiation Thermometer
Measurement scatter	0.4j.iV	Normal	0.24j.iV	1	0.24	maximum difference of EMF in 3 time measurements
Inflection point calculation	± 0.1 j.iV	Rectangular	0.06j.iV	1	0.06	variation of calculation method
Conduction Errors	±0.2°C	Rectangular	0.12°C	14.1	1.69	temperature difference between cell and thermocouple
Inhomogeneity						
CJ temperature	±0.02°C	Rectangular	0.012°C	5.3	0.06	impurity of water and conduction of stainless steel tube
DVM calibration	0.2j.iV	Normal	0.1j.iV	1	0.10	calibration uncertainty at 100 mV range
DVM Short-term stability	± 0.67j.iV	Rectangular	0.39j.iV	1	0.39	90 Day accuracy 25ppm*reading+3ppm*range
Stray EMF /Electric noise	± 0.1j.iV	Rectangular	0.06j.iV	1	0.06	AC pick up and electric leakage of heater
Rounding Error	± 0.1j.iV	Rectangular	0.06j.iV	1	0.06	Rounded to the nearest 0.1j.iV
Combined standard uncertainty /uV						

Expanded uncertainty (k = 2) /uV	5.48
Expanded uncertainty (k = 2) /ºC	0.39

NIM, China

Uncertainty Analysis of R Type thermocouple (2017-05) at Palladium melting Point

Unceratiny factors	Quantity	Probability Distribution	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution /j.iV
erature Value of Palladium	n±0.28°	Normal	0.14°C	13.98	1.96
metal quality	±0.3°C	Rectangular	0.17°C	13.98	2.42
Measurement scatter	1.7j.iV	Normal	1.0j.iV	1	1.00
plateau determination	± 0.5j.iV	Rectangular	0.29j.iV	1	0.29
Conduction Errors	±0.3°C	Rectangular	0.17°C	13.98	2.42
Inhomogeneity					0.00
CJ temperature	±0.02°C	Rectangular	0.012°C	5.3	0.06
DVM calibration	0.2j.iV	Normal	0.10 j.iV	1	0.10
DVM Short-term stability	± 0.76j.iV	Rectangular	0.44j.iV	1	0.44
Stray EMF /Electric noise	± 0.2j.iV	Rectangular	0.12j.iV	1	0.12
Rounding Error	± 0.1j.iV	Rectangular	0.06j.iV	1	0.06
			Combined standard uncertainty /uV		

Expanded uncertainty (k = 2) /uV	8.21
Expanded uncertainty (k = 2) /°C	0.59

Remarks
melting point temperature of Palladium in Argon impurity of Pd wire and Pd oxidized in melting procedure maximum difference of EMF in 3 time measurements determintation of melting Plateau temperature gradient in Radial and Axial direction impurity of water and conduction of stainless steel tube calibration uncertainty at 100 mV range 90 Day accuracy 25ppm*reading+3ppm*range AC pick up and electric leakage of heater Rounded to the nearest 0.1μV

Type R 2017-07, NIMT, Thailand

2nd round

Type	Source of uncertainty	Uncertainty value	Probability distribution	Divisor	Sensitivity coefficient (c_i)	Standard uncertainty (uV)	Degrees of freedom
A	Statistical standard uncertainty (uV)	0.07	normal	1	1.0	0.0699	2
B	Inhomogeneity of UUC (uV) 0.0xx %E	0.00	rectangular	SQRT(3	1.0	0.0000	□
B	Calibration of fixed point cel (°C)	0.05	normal	2	13.6	0.3191	□
B	Correction due to the calibration of fixed point cel (°C	0.00	rectangular	SQRT(3	1.0	0.0000	□
B	Calibration of emf indicator (uV)	0.10	normal	2	1.0	0.0500	□
B	Voltage correction due to the contact (uV)	0.30	rectangular	SQRT(3	1.0	0.1732	□
B	Long term drift of voltmeter (uV)	0.50	rectangular	SQRT(3	1.0	0.2887	□
B	Resolution of emf indicator (uV)	0.01	rectangular2*SQRT(3		1.0	0.0029	□
B	Heat conduction error (uV)	1.00	rectangular	SQRT(3	1.0	0.2887	□
B	Uniformity of constant temperature ice bath (°C)	0.01	rectangular	SQRT(3	13.6	0.0784	□
							□
-	Combinded standard uncertainty		normal			0.5586	> 200
-	Expanded uncertainty		normal (k= ...	C	0.1	1.1172	2.00

Calibration Point, (°C) = 1084.620

Type	Source of uncertainty	Uncertainty value	Probability distribution	Divisor	Sensitivity coefficient (c_i)	Standard uncertainty (uV)	Degrees of freedom
A	Statistical standard uncertainty (uV)	0.10	normal	1	1.0	0.1008	2
B	Inhomogeneity of UUC (uV) 0.02 %E	0.00	rectangular	SQRT(3	1.0	0.0000	□
B	Calibration of fixed point cel (ºC)	0.44	normal	2	14.1	3.1020	□
B	Plateau determination of fixed point cell (uV)	1.00	rectangular	SQRT(3	1.0	0.5774	□
B	Calibration of emf indicator (uV)	0.10	normal	2	1.0	0.0500	□
B	Voltage correction due to the contact (uV)	0.30	rectangular	SQRT(3	1.0	0.1732	□
B	Long term drift of voltmeter (uV)	0.50	rectangular	SQRT(3	1.0	0.2887	□
B	Resolution of emf indicator (uV)	0.05	rectangular	SQRT(3	1.0	0.0144	□
B	Heat conduction error (uV)	1.00	rectangular	SQRT(3	1.0	0.2887	□
B	Uniformity of constant temperature ice bath (ºC)	0.01	rectangular	SQRT(3	14.1	0.0814	□
-	Combinded standard uncertainty		normal			3.189	> 200
-	Expanded uncertainty		normal (k= ...	C	0.5	6.379	2.00

Calibration Point, (ºC) = 1324.06

NIMT, Thailand

Type	Source of uncertainty	Uncertainty value	Probability distribution	Divisor	Sensitivity coefficient (cI)	Standard uncertainty (uV)	Degrees of freedom
A	Statistical standard uncertainty (uV)	0.20	normal	1	1.0	0.200	2
B	Inhomogeneity of UUC (uV) 0.02%E	0.00	rectangular	SQRT(3	1.0	0.000	∞
B	Bridge wire impurity (ºC)	0.30	normal	1	14.0	4.200	∞
B	Plateau determination of fixed point cell (uV)	5.00	rectangular	SQRT(3	1.0	2.887	∞
B	Calibration of emf indicator (uV)	0.10	normal	2	1.0	0.050	∞
B	Voltage correction due to the contact (uV)	0.30	rectangular	SQRT(3	1.0	0.173	∞
B	Long term drift of voltmeter (uV)	0.50	rectangular	SQRT(3	1.0	0.289	∞
B	Resolution of emf indicator (uV)	0.05	rectangular	SQRT(3	1.0	0.029	∞
B	Heat conduction error (uV)	5.00	rectangular	SQRT(3	1.0	2.887	∞
B	Uniformity of constant temperature ice bath (ºC)	0.01	rectangular	SQRT(3	14.0	0.081	∞
-	Combinded standard uncertainty		normal			5.871	> 200
-	Expanded uncertainty		normal (k= ...	C	0.8	11.742	2.00

Calibration Point, (ºC) = 1553.50

NMIJ, Japan

Uncertainty Analysis

Source of uncertainty	Co-C point /µV	Pd point /µV
Uncertainty of the fixed point temperature	3.69	3.22
Measurement scatter at the fixed point	0.10	0.21
Conduction errors	0.05	0.00
Uncertainty due to inhomogeneity of thermocouple		
CJ temperature	0.01	0.01
DVM calibration and its use	0.18	0.19
Rounding/ Resolution error	0.003	0.03
Stray EMFs and electrical noise	0.01	0.05
Combined standard uncertainty	3.70	3.23
Expanded uncertainty (*k* = 2) in µV	7.4	6.5

Laboratory Name: National Metrology Institute of South Africa (NMISA)

REF STD: Cu29056 (open)	Value	Unit	Divisor	Sensitivity coeff	ui(k=1) (°C)	D.o.f.	
Fixed pt:							
Chemical impurity:	0.00087	°C	1.732	1	**0.001**	500	1.28E-16
Fixed pt realisation:	0.00242	µV	1.732	0.07366	**0**	958	1.18E-19
Gas pressure	15	Pa	1.732	3.30E-08	**0**	500	1.33E-29
Hydrostatic head	0.04662	m	1.732	2.60E-03	**0**	500	4.80E-20
UUT:							
Thermoelectric inhomogeneity		V/V	1.732		**0**	500	0.00E+00
Uncert of ref temp	0.088	°C	1.732	0.38966	**0.02**	500	3.12E-10
Cal of meas. instrument	0.26274	µV	2	0.07366	**0.01**	500	1.75E-11
Spec of meas. instrument	0.62186	µV	1.732	0.07366	**0.026**	500	9.79E-10
Spurious thermal emfs	0.4	µV	1.732	0.07366	**0.017**	500	1.68E-10
Compensating wire cold junction	0.05	°C	1.732	1	**0.029**	500	1.39E-09
Stem conduction	0.00749	°C	1.732	1	**0.004**	500	6.99E-13
Repeatability	0.00085	µV	1	0.07366	**0**	345	4.52E-20
Reproducibility	0.3385	µV	1	0.07366	**0.025**	2	1.93E-07
uc(k=1) (°C):					**0.054**		
Eff deg of freedom					**44.409**		**1.96E-07**
t(eff d.o.f.):					**2.06**		
U(k=2) (°C):					**0.112**		
U(k=2) (µV):					**1.52**		

Laboratory Name: National Metrology Institute of South Africa (NMISA)

REF STD: Pd_wire bridge method	Value	Unit	Divisor	Sensitivity coeff	ui(k=1) (°C)	D.o.f.	
Fixed pt:							
Chemical impurity:	0.0001	mol/mol	1.732	1661.13	**0.096**	500	1.69E-07
Fixed pt realisation:	0.05353	µV	1	0.071524	**0.004**	1	2.15E-10
O2 pressure	1.61245	√kPa	1.732	-0.375	**-0.349**	500	2.97E-05
UUT:							
Thermoelectric inhomogeneity		V/V	1.732	1291.844	**0**	500	0.00E+00
Uncert of ref temp	0.088	°C	1.732	0.378333	**0.019**	500	2.77E-10
Cal of meas. instrument	0.39391	µV	2	0.071524	**0.014**	500	7.88E-11
Spec of meas. instrument	1.30977	µV	1.732	0.071524	**0.054**	500	1.71E-08
Spurious thermal emfs	0.4	µV	1.732	0.071524	**0.017**	500	1.49E-10
Compensating wire cold junction	0.05	°C	1.732	1	**0.029**	500	1.39E-09
Stem conduction	0.0199	°C	1.732	1	**0.011**	500	3.49E-11
Repeatability	1.3935	µV	1	0.071524	**0.1**	1	9.87E-05
Reproducibility	4.64184	µV	1.732	0.071524	**0.192**	2	6.75E-04
uc(k=1) (°C):					**0.427**		
Eff deg of freedom					**41.447**		**8.04E-04**
t(eff d.o.f.):					**2.06**		
U(k=2) (°C):					**0.88**		
U(k=2) (µV):					**12.3**		

Name of the Laboratory : NPLI India

Appendix D : Uncertainty of Measurement of the Thermocouple **Serial Number : 2017-02**

Fixed Point : FP of Cu (1084.62 oC)

Source of Uncertainty	Quantity (1V) Distribution	Probability function/Type	Standard uncertainty (µV)	Sensitivity coefficient	Uncertainty contribution / (µV)	Degree of Freedom	Remarks
Uncertainty of the fixed point Temperature	2.80	Normal! Type-B	1.62	1	1.62	∞	Factors related to realization of FP of Cu by thermocouple
Measurement Scatter at the fixed points	0.26	Normal! Type-A	0.15	1	0.15	2	Standard deviation of 3 runs
Conduction Errors	0.16	Rectangular! Type-B	0.09	1	0.09	∞	Change in measured emf for 2 cm dispaclement of thermocouple
Uncerainty due to inhomogneity of the thermocouple	Excluded						To be provided by Pilot Laboratory
CJ temperature	0.03	Rectangular! Type-B	0.01	1	0.01	∞	Estimated!certificate
DVM Calibration	0.11	Normal!Type-B	0.06	1	0.06	∞	Certificate
Rounding! Resolution error	0.01	Rectangular! Type-B	0.00	1	0.00	∞	0.01 1iV resolution of DVM, 0.007 1iV rounding-off
Stray EMFs and elctrical Noise	0.03	Rectangular! Type-B	0.02	1	0.02	∞	Estimate on stray noise
Combined Standard Uncertainty! 1iV					1.63		
Expanded uncertainty ! 1iV (*k* =2)					3.25		µV
Expanded uncertainty ! oC (*k* =2)					0.23		oC

Name of the Laboratory : NPLI India Serial Number : 2017-02

Appendix D : Uncertainty of Measurement of the Thermocouple

Fixed Point : MP of Co-C (1323.93 oC)

Source of Uncerainty	Quantity (i.L	Probability Distribution	Standard uncertainty (μV)	Sensitivity coefficient	Uncertainty contribution / (μV)	Degree of Freedom	Remarks
Uncertainty of the fixed point Temperature	5.00	Normal/ Type-B	2.50	1	2.50	∞	Factors related to realization of MP of Co-C by radiation thermometry and thermocouple, fixed point made-up of same Co-C content/batch
Inflection Point	0.48	Rectangular/ Type-B	0.28	1	0.28	∞	3 runs, (max-min)/2
Measurement Scatter at the fixed points	0.49	Normal/ Type-A	0.28	1	0.28	2.00	Standard deviation of 3 runs
Conduction Errors	0.50	Rectangular/ Type-B	0.29	1	0.29		Change in measured emf for 2 cm dispalcement of thermocouple
Unceraintiy due to inhomogneity of the thermocoup	eExclude					∞	
CJ temperature	0.03	Rectangular/ Type-B	0.01	1	0.01	∞	Estimated/certificate
DVM Calibration	0.11	Normal/ Type-B	0.06	1	0.06	∞	Certificate
Tc Stability	0.56	Rectangular/ Type-B	0.32	1	0.32	∞	EMF difference in Cu before and after
Rounding/ Resolution error	0.01	Rectangular/ Type-B	0.004	1	0.004	∞	0.01 i.LV resolution of DVM, 0.007 i.LV rounding-off
Stray EMFs and elctrical Noise	0.03	Rectangular/ Type-B	0.02	1	0.02	∞	Estimate on stray noise
Combined standard Uncertainty/ i.LV					2.57		
Expanded uncertainty / i.LV (*k* =2)					5.14		μV
Expanded uncertainty / oC (*k* =2)					0.37		oC

Name of the Laboratory : NPLI India Serial Number : 2017-02

Appendix D : Uncertainty of Measurement of the Thermocouple

Fixed Point : MP of Pd (1554.8 oC) by wire-bridge

Source of Unceraincy	Quantity (µV)	Probability Distribution	Standard uncertainty (µV)	Sensitivity coefficient	Uncertainty contribution / (µV)	Degree of Freedom	Remarks
Uncertainty of the fixed point Temperature	5.86	Normal/Type-B	2.93	1	2.93	∞	Factors related to realization of MP of Pd by wire-bridge using thermocouple
Measurement Scatter at the fixed points	0.80	Normal/Type-A	0.46	1	0.46	2	Standard deviation of 3 runs
Conduction Errors	0.00	Rectangular/ Type-B	0.00	1	0.00	∞	Wire-bridge method
Unceraincy due to inhomogneity of the thermocouple	Excluded						
CJ temperature	0.03	Rectangular/ Type-B	0.01	1	0.01	∞	Estimated/certificate
DVM Calibration	0.11	Normal/ Type-B	0.06	1	0.06	∞	Certificate
Tc Stability	1.20	Rectangular/ Type-B	0.69	1	0.69	∞	EMF difference in Cu before and after Pd points
Rounding/ Resolution error	0.01	Rectangular/ Type-B	0.004	1	0.004	∞	0.01 liV resolution of DVM, 0.007 liV rounding-off
Stray EMFs and elctrical Noise	0.03	Rectangular/ Type-B	0.02	1	0.02	∞	Estimate on stray noise
Combined standard Uncertainty/ liV					3.05		
Expanded uncertainty / liV (*k* =2)					6.09		liV
Expanded uncertainty / oC (*k* =2)					0.44		oC

Name of Laboratory: KRISS

Uncertainty calculation Serial Numbe2017-10

Uncertainty components	FP	Quantity	Probability distribution	Standard uncertainty	Sensitivity coefficient	Uncertainty contribution /µV	Remarks
Uncertainty of the fixed point tempertaure	Cu	0.016 °C	Normal	0.008 °C	13.58 µV/°C	0.11	claimed at APMP.T-S1
	Co-C	0.8 °C	Normal	0.4 °C	14.10 µV/°C	5.64	Type R reference thermocouple calibrate radiation thermometer
	Pd	1.2 °C	Normal	0.6 °C	13.97 µV/°C	8.38	Type B reference thermocouple calibrate radiation thermometer
Measurement scatter at the fixed point	Cu	0.05 µV	Normal	0.05 µV	1	0.05	Standard deviation
	Co-C	0.18 µV	Normal	0.18 µV	1	0.18	
	Pd	0.45 µV	Normal	0.45 µV	1	0.45	
Conduction errors	Cu	0.1 µV	Rectangular	0.06 µV	1	0.06	
	Co-C	0.1 µV	Rectangular	0.06 µV	1	0.06	
	Pd	0.2 µV	Rectangular	0.12 µV	1	0.12	
Uncertainty due to Inhomogeneity	Cu						
	Co-C						
	Pd						
CJ temperature	Cu	10 mK	Rectangular	5.6 mK	13.58	0.08	
	Co-C	10 mK	Rectangular	5.6 mK	14.10	0.08	
	Pd	10 mK	Rectangular	5.6 mK	13.97	0.08	
DVM calibration and its use	Cu	0.06 µV	Normal	0.06 µV	V/°C 1	0.06	calibration uncertainty µV/V (*k* =2)
	Co-C	0.07 µV	Normal	0.07 µV	1	0.07	
	Pd	0.09 µV	Normal	0.09 µV	1	0.09	
DVM long-term stability	Cu	0.23 µV	Rectangular	0.13 µV	1	0.13	Long-term stability: 20
	Co-C	0.30 µV	Rectangular	0.17 µV	1	0.17	
	Pd	0.37 µV	Rectangular	0.21 µV	1	0.21	
Rounding/ Resolution error	Cu	0.01 µV	Rectangular	0.003 µV	1	0.003	
	Co-C	0.1 µV	Rectangular	0.03 µV	1	0.03	
	Pd	0.1 µV	Rectangular	0.03 µV	1	0.03	
Stray EMFs and electrical Noise	Cu	0.1 µV	Rectangular	0.03 µV	1	0.03	
	Co-C	0.1 µV	Rectangular	0.03 µV	1	0.03	
	Pd	0.1 µV	Rectangular	0.03 µV	1	0.03	
Combined standard uncertainty (*k* =1)	Cu					0.21	
	Co-C					5.65	
	Pd					8.40	
Expanded uncertainty (*k* =2)	**Cu**					**0.43**	
	Co-C					**11.3**	
	Pd					**16.8**	