Traceable Thermocouple Calibration in RCM-LIPI

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Abstract. This article reports current status and future plans of thermocouple thermometry in RCM-LIPI. It depicts calibration facilities, measurement capabilities, and traceability in the lab. Recommendations have been made for the improvement of thermocouple thermometry scale, especially for the scale at a temperature range above 1100 °C. The recommendations are about participation in international laboratory comparison (ILC), construction of eutectic fixed points, and validation of the scale against radiation thermometry scale.

Keywords: traceability; thermocouple; calibration;

1. Introduction
Thermal processing plays a decisive role during numerous industrial processes, e.g., hardening (1, 2), tempering (3), and annealing (4). These processes require a thermal sensor with a characteristic of wide measurement range and fast response. In almost cases, thermocouples are selected as the sensor and they are routinely calibrated to ensure their accuracy. Calibration of thermocouple consists of several steps, which include: establishing, maintaining, and disseminating the measurement standards. As a national metrology institute (NMI) in Indonesia, Research Center for Metrology (RCM-LIPI) is liable for these steps.

RCM-LIPI establishes the measurement standard of thermocouple by the realization of the defining fixed-points according to the international temperature scale of 1990 (ITS-90) using noble-metal thermocouple as the interpolating instruments (5). The fixed-points are divided into two levels: primary and secondary. In order to establish the scale in the range of 0.01 °C to 1084.62 °C for thermocouple thermometry, several primary fixed-points are needed: triple-point (TP) of water, freezing-points (FP) of Sn, Zn, Al, Ag, and Cu (5). Furthermore, to extend the scale up to 1553.5 °C, a secondary level fixed-point, the melting point of Pd wire in air, is used as the reference (6).

RCM-LIPI maintains the measurement standard by performing intermediate check and internal comparison of fixed-point cells. To support these processes, the cells are created redundant (7). RCM-LIPI also joins international comparisons to ensure the traceability and reliability of the measurement standard. The results of these comparisons are listed in Appendix C, CMC of the certificate in investment performance measurement - mutual recognition arrangement (CIPM - MRA) (8).

RCM-LIPI disseminates the measurement standard of thermocouple by the calibration scheme. The calibration method is a comparison against the noble-metal thermocouple (transfer standard) obtained from the realization of ITS-90 scale. This method covers the calibration range from 0.01 °C to 1553.5 °C.

In this article, calibration facilities, measurement capabilities, traceability scheme, and future plans of thermocouple thermometry in RCM-LIPI are reported. This article will be important not only to
explain the current status of thermocouple thermometry in RCM-LIPI but also to give design route of thermocouple thermometry improvement for other developing NMIs.

2. Calibration Facilities

2.1 Calibration against the Defining Fixed-Points

2.1.1 Triple point of water cell

So far, RCM-LIPI has owned seven triple point of water (TPW) cells: five have been obtained from other NMIs (PTB and MSL) and two have been constructed at RCM-LIPI. Fig. 1 shows the realization of TPW in RCM-LIPI. TPW is realized using the frozen carbon dioxide (dry ice) method. The measurement well of TPW is filled with dry ice to create an ice mantle. After reaching the triple-point, the cell is kept in a maintenance bath or in a Dewar flask filled with melting ice to prolong the plateau.

![Fig. 1](image)

Fig. 1 (a) filling dry ice into the measurement well of TPW cell to create (b) the ice mantle. (c) After the cell having three phases of water, it is maintained in (d) a Dewar flask filled with melting ice.

2.1.2. Freezing point of pure metal

A list of fixed-points used in RCM-LIPI to realize the scale of thermocouple thermometry is shown in Table 1. RCM-LIPI uses commercially available Hart Scientific fixed-point cells and also develops their own fixed-point cells. The purity of metal inside the cells is about 99.9999% (6N). The pure metal is contained in a graphite crucible (with the purity and the density are greater than 99.999% and 1.9 g-cm$^{-3}$, respectively) and then the crucible is kept in a quartz crucible. Afterward, the quartz crucible is placed in a vertically-homogenous temperature furnace where the vertical temperature gradient should not exceed about ±10 mK (9). The cells are open type where the pressure inside the cell can be adjusted by filling with ultra-high purity (99.9999%) argon gas.

| No. | Fixed-Point Cell | Manufacturer, Purity | Immersion Depth (mm) |
|-----|------------------|----------------------|----------------------|
| 1   | Sn FP            | Hart Scientific, 6N  | 170                  |
| 2   | Zn FP            | Hart Scientific, 6N  | 170                  |
| 3   | Al FP            | Hart Scientific, 6N  | 170                  |
| 4   | Al FP            | RCM-LIPI, 6N         | 170                  |
| 5   | Ag FP            | Hart Scientific, 6N  | 170                  |
| 6   | Cu FP (new cell) | RCM-LIPI, 6N         | 170                  |

The cells are mounted into a vacuum system and argon filling system using stainless-steel piping system as shown in Fig. 2. The system is sufficient to simultaneously accommodate five fixed-point cells. The vacuum system uses the combination of mechanical and turbo-molecular pumps to achieve
the pressure in the cell to be less than $10^{-6}$ Torr. The air-pumping process shall not be done together with the argon-filling process. Hence, several valves are used to control the flow of air in the system. A pressure regulator is equipped in an argon gas cylinder to reduce the input pressure of the gas to a desired level at its output toward the cell. Two valves located in between argon gas cylinder and fixed-point cells are used to set the pressure in the cell. The pressure is then measured using a calibrated pressure gauge.

![Fig. 2 Schematic diagram of freezing metal fixed-points system in RCM-LIPI](image)

The realization of FP of pure metal is almost identical for all fixed-points. First, the cell is pre-heated for at least 2 hours at a temperature far below its melting temperature while it is also continually pumped using a turbo-molecular pump. Second, after pre-heating, the cell temperature is set at a temperature slightly higher than its melting temperature (2 °C to 5 °C above melting point). Third, when the pure metal ingot reaches the melting point, the pressure in the cell is adjusted to be atmospheric. Fourth, the ingot in the cell is melted overnight. Fifth, the melted ingot is then allowed to cool down slowly to a temperature lower than FP temperature, at a rate of about 0.1 °C/min and the ingot temperature will drop to the recalescence temperature (several degrees below its freezing temperature). Sixth, soon after the recalescence temperature is observed, the monitoring thermometer is withdrawn and a room-temperature quartz-rod is inserted into the cell for several minutes to allow inner nucleation. Seventh, after thermal stabilization, the cell is ready for measurement.

2.1.3. Melting palladium wire point

The melting point of Pd wire is realized using ‘mini-coil’ method in accordance with the procedure reported by Jahan, F and Ballico, M (10). Fig. 3 shows a schematic diagram of the realization of the melting point of Pd using mini-coil method. A 0.5-mm-diameter Pd wire 0.5 mm having a nominal purity of 99.99 % is used to create the coils. The coils are then cleaned by boiling in HCL and distilled water solution.

![Fig. 3 Schematic diagram of melting Pd wire realization using mini-coil method](image)
Thermocouple wires are pulled about 10 mm from the ceramic insulator to attach the coil. The wires are cut at the junction and the coil is inserted into one leg of the thermocouple wires. The wires are then connected by welding using non-contaminating hydrogen torch. The coil position is set so that the middle of the coil is at the thermocouple junction (see Fig. 3). The wires are then pulled back to the ceramic insulator and the insulator is then inserted into a one-closed end ceramic tube.

The thermocouple is inserted into the annealing furnace (1600B, Land, Dronfield, UK). The furnace temperature is set at about 8 °C below the melting point of Pd (1545.5 °C). After thermal stabilization, the furnace temperature is ramped to a temperature approximately 5 °C above the melting point of Pd (1558.5 °C) with a ramp-rate between 0.2 °C/min and 2 °C/min. As soon as the melting point temperature is reached, the temperature slope suddenly decreases where this is the indication of the beginning of the melt.

2.2 Calibration by Comparison against Reference Thermometers

Apart from the calibration of thermocouple by fixed-points, the calibration of thermocouple also can be done by comparison against reference thermometers (standard platinum resistance thermometers (SPRTs) and noble-metal thermocouple). Normally, a base-metal thermocouple is calibrated against a noble metal thermocouple at a temperature range between 0.01 °C and 1553.5 °C. However, for a temperature range between -38.8344 °C and 961.78 °C, the best CMC can be obtained when the reference thermometer is SPRT. For a higher temperature range, between 961.78 °C and 1553.5 °C, SPRT cannot be used as the reference thermometer due to the limitation of the quartz protecting tube used.

3. Traceability and Measurement Capabilities

Measurement traceability of thermocouple thermometry to the SI units is established by the participation in international laboratory comparisons (ILCs). This section reports the participation of RCM-LIPI in ILCs to date and the measurement capabilities of thermocouple thermometry established in RCM-LIPI.

3.1 International Comparison Participations

3.1.1 TPW cell

In 2007-2009, RCM-LIPI participated in an international key comparison of TPW cells, APMP.T-K7. In this comparison, one cell was sent to the centre for measurement standards (CMS), NMI of Chinese Taipei. The TPW cell was made of borosilicate glass with the cell dimensions are as follows: the outer cell diameter is 50 mm, the inner cell diameter is 12 mm, and the depth of the thermometer well is 215 mm. The final report of this comparison shows that RCM-LIPI measurement at TPW point is in good agreement (within 58.8 µK) with the Asia pacific metrology program (APMP) reference value (11). The value of the comparison is disseminated to the secondary reference TPW cell and this cell is then used as a standard for calibration of working standard, i.e., in the calibration of noble metal thermocouple.

3.1.2 Hg to Zn Fixed-Points

RCM-LIPI participated in regional and bilateral comparisons of fixed points. These comparisons can be used as a link to the SI units for thermocouple thermometry by the use of calibrated SPRT (to the linked fixed-points) as a reference thermometer. The regional comparison was initiated by the Asia-Pacific Metrology Program (APMP) entitled APMP-K3: Key Comparison of Realizations of the ITS-90 over the Range -38.8344 °C to 419.527 °C (12). The bilateral comparison was initiated by the request of RCM-LIPI to the Korea Research Institute of Standards and Sciences (KRISS) entitled APMP.T-K3.4: Key Comparison of Realizations of the ITS-90 over the Range -38.8344 °C to 419.527 °C (13).

For APMP-K3, the artifact of comparison was a 25.5 Ω SPRT (419, Isotech, Southport, UK). The artefact was circulated using loop mechanism to 12 participants. The measurements were conducted
from February 2000 to June 2003. Although this comparison covers a temperature range between triple point (TP) of Hg to FP of Zn, RCM-LIPI joined this comparison for measurement of SPRT only at FP of Sn and Zn. Unfortunately, the results were not satisfactory (outlier) for those points because the uncertainty of RCM-LIPI’s measurement was not able to cover the difference between RCM-LIPI’s value and the average reference value (ARV) of the CCT-KC3. The difference between RCM-LIPI’s value and ARV of CCT-KC3 for FP of Sn and Zn was 2.7 mK (at \( U=2.1 \) mK) and 2.9 mK (at \( U=1.5 \) mK), respectively. In order to get more satisfying results, RCM-LIPI was then requested the bilateral comparison with KRISS for linking the CCT-KC3-ARV values of Hg to Zn fixed-points.

For APMP-T-K3.4, the artifacts of comparison were two 25.5 Ω SPRTs (670SQ, Isotech, Southport, UK). The artifacts were measured in a RCM-LIPI—KRISS—RCM-LIPI sequence. The artifacts were measured at TP of Hg, melting point of Ga, FP of In, Sn, and Zn. The measurements were conducted from 2011 to 2013. RCM-LIPI’s measurements were in good agreement (inlier) with the ARVs of the CCT-K3. The highest difference was observed in FP of Zn (\(-5\) mK at \( U=10\) mK).

### 3.1.3 Type R (Pt-Pt13%Rh) Thermocouples

RCM-LIPI participated in Regional Comparison of Type R (Pt-Pt13% Rh) Thermocouples from 0 to 1100°C, APMP.T-S1 in 2004 (8). Pilot laboratory of this comparison was the National Measurement Institute of Australia (NMIA). 12 participants from the Asia Pacific NMIs including pilot laboratory took part in this comparison. The artifact was 11 type R thermocouples named APMP-01 to APMP-11. The artifacts were circulated to participants using a star type comparison. The measurements were done from March to November 2005. Each participant was free to choose the method they used to calibrate the thermocouple.

RCM-LIPI used furnace calibration against SPRT—for temperature below 400 °C—and a type S thermocouple—for temperature above 400 °C. RCM-LIPI’s results of this comparison were satisfactory. From 17 measurement points, there is only one outlier point (at 0 °C). The highest difference (\(-3.068\) µV, \( U=4\) µV) was observed at the calibration point of 1100 °C.

### 3.2 Measurement Capabilities

#### 3.2.1 Fixed point method

The uncertainty of the calibration of type S thermocouple against fixed-point cells (from TPW to FP of Cu) is summarized in Table 1. Uncertainty due to fixed-points realization is obtained from the latest CMC value of the respective cells. The long-term stability is estimated to be within 0.001 °C for all fixed-points. Thermocouple wire inhomogeneity is estimated between 0.02 and 0.03%. The repeatability of measurement is calculated from 20 times emf reading during cell plateau. The use of ice-point at the reference junction of thermocouple contributes 0.012 °C of standard uncertainty. The digital voltmeter (DVM) used is calibrated to the electrical quantity of voltage laboratory and the uncertainty reported is included in the table. DVM drift is estimated from the maximum difference from two latest DVM calibration certificate. The three most common noise sources in voltage measurement are the common mode, normal mode, and electrostatic. The total noise (summation of these three noises) contributes about 0.2 µV of expanded uncertainty into the measurement system.

Parasitic emf is estimated to add 0.48 µV of expanded uncertainty to the total uncertainty. The rounding is estimated to contribute about 0.05 µV of expanded uncertainty.

#### Table 1 Uncertainty in the calibration of type S thermocouple by fixed-points method (TPW to FP of Cu)

| No.  | Uncertainty Sources                  | Standard Uncertainty (k=1), °C |
|------|-------------------------------------|-------------------------------|
|      |                                     | TPW  | Sn     | Zn     | Al     | Ag     | Cu     |
| 1    | Fixed-point realization             | 0.001| 0.002  | 0.002  | 0.003  | 0.003  | 0.008  |
| 2    | Long-term stability                 | 0.001| 0.001  | 0.001  | 0.001  | 0.001  | 0.001  |
| 3    | Thermocouple wire inhomogeneity     | 0.003| 0.020  | 0.039  | 0.062  | 0.090  | 0.151  |
Apart from the common open fixed-point cell uncertainty budget, the uncertainty budget of calibration of type S thermocouple by melting Pd wire is shown in Table 2. The uncertainty components due to repeatability, DVM certificate, DVM drift, thermocouple wire inhomogeneity, parasitic emf, the use of ice-point, and rounding error are obtained in the same way as for the calibration in open fixed-point cell. The purity of Pd wire contributes about 0.1 °C of expanded uncertainty to the measurement system where is in the same order of magnitude with the value reported by Jahan, F and Ballico, M (10). The uncertainty due to pressure effect arises as the system pressure cannot be controlled. The standard uncertainty value of pressure effect is about 0.001 °C. The present of oxygen and melting point realization add standard uncertainty about 0.115 °C and 0.213 °C, respectively, to the system. The standard uncertainty due to reproducibility of melting Pd wire realization is 0.213 °C, determined from the standard deviation of measured emf of three plateaus. Thermal contact between Pd wire and thermocouple wire contributes 0.098 °C of standard uncertainty to the system.

Table 2 Uncertainty in the calibration of type S thermocouple by melting Pd wire method

| No. | Uncertainty Sources                        | Standard Uncertainty \((k=1)\), °C |
|-----|-------------------------------------------|----------------------------------|
| 1   | Purity of Pd wire                         | 0.058                            |
| 2   | Pressure effect                           | 0.001                            |
| 3   | The present of oxygen                     | 0.115                            |
| 4   | Melting point realization                 | 0.213                            |
| 5   | Reproducibility                           | 0.006                            |
| 6   | Repeatability of measurement              | 0.272                            |
| 7   | DVM certificate                           | 0.006                            |
| 8   | DVM drift                                 | 0.020                            |
| 9   | The use of ice-point                      | 0.049                            |
| 10  | Thermocouple wire inhomogeneity           | 0.230                            |
| 11  | Thermal contact                           | 0.098                            |
| 12  | Parasitic emf                             | 0.001                            |
| 13  | Rounding error                            | 0.002                            |
|     | Combined standard uncertainty \((k=1)\), °C | 0.90                             |

3.2.2 Comparison method

Table 3 shows the uncertainty budget of type S thermocouple calibration by comparison method. The uncertainty components due to DVM certificate (DVM reading), DVM drift, parasitic emf, the use of ice-point, electrical noise, and rounding error are obtained in the same way as for the calibration in open fixed-point cell. The standard thermocouple is calibrated against RCM-LIPI’s fixed-points. The drift of standard thermocouple is determined from the mid-range of maximum different in correction between two latest it calibration certificates at each temperature. Inhomogeneity of thermocouple...
under test is measured from tip to 400 mm at 200 °C in an oil bath (14). The uncertainty due to calibration furnace for a calibration range below and above 1084.62 °C is measured to be 0.048 °C and 0.081 °C, respectively. Most common noble metal thermocouple sensors are equipped with an extension wire. However, in some cases, when the calibrated thermocouple comes without an extension wire, two sets of copper wire are used as an extension wire. Based on our characterization, these extension wires contribute about 0.037 °C of expanded uncertainty to the measurement system. When reporting the correction as a function of temperature \( f(t) \), the standard error estimate (SEE) of the best fit plot has to be reported as one of the uncertainty sources. The SEE is obtained from regression analysis in MS-Excel.

### Table 3 Uncertainty in the calibration of type S thermocouple by comparison method for a respective calibration range

| No. | Uncertainty Sources                                      | Standard Uncertainty \((k=1), °C\) |
|-----|---------------------------------------------------------|-----------------------------------|
|     |                                                         | (0.01-1084.62) °C (1084.62-1553.5) °C |
| 1   | Certificate of standard thermocouple                    | 0.03+0.000001*\(t\) 0.538+0.000067*\(t\) |
| 2   | Drift of standard thermocouple                          | 0.060 0.375 |
| 3   | Inhomogeneity of thermocouple under test                | 0.00012*\(t\) 0.00029*\(t\) |
| 4   | Non-uniformity and instability of furnace               | 0.048 0.081 |
|     | The use of ice-point for thermocouple standard           | |
| 5   | The use of ice-point for thermocouple under test         | 0.012 0.012 |
| 6   | DVM reading of standard thermocouple                    | 0.012 0.012 |
| 7   | DVM reading of thermocouple under test                  | 0.005 0.006 |
| 8   | DVM drift                                               | 0.005 0.005 |
| 9   | Electrical noise                                        | 0.029 0.017 |
| 10  | Parasitic emf                                           | 0.007 0.006 |
| 11  | Ext. wire of thermocouple standard                       | 0.030 0.001 |
| 12  | Ext. wire of thermocouple under test                     | 0.021 0.021 |
| 13  | DVM drift                                               | 0.000 0.021 |
| 14  | Rounding error                                          | 0.002 0.002 |
| 15  | SEE                                                     | 0.047 0.020 |
|     | Combined standard uncertainty \((k=1), °C\)             | 0.11+0.00012*\(t\) 0.66+0.00030*\(t\) |

### 4. Conclusions and Future Plans

Some parts of measurement capabilities of thermocouple thermometry in RCM-LIPI were already internationally recognized. For calibration of thermocouple at a range of \(-38.8344 \, ^\circ C\) to \(1100 \, ^\circ C\), the measurement traceability to the SI units can be drawn. However, for calibration of thermocouple at a range above \(1100 \, ^\circ C\), the traceability is not yet established. To solve this problem, RCM-LIPI is joining the APMP.T-S16: the APMP Regional Comparison of Type R Thermocouples Above \(1100 \, ^\circ C\). The latest update of this comparison is still in the progress of protocol preparation. Another issue to address would be the lack of number of standard for the establishment of thermocouple scale above \(1100 \, ^\circ C\). This issue can be solved by the construction of eutectic standards or by validation of the scale against radiation thermometry scale.

### Acknowledgments

This work is supported by Ministry of Research, Technology and Higher Education of the Republic of Indonesia through the scheme of Incentive Research Program for the National Innovation System.
We acknowledge the insights gained while working with A. Achmadi and G. Zaid.

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