# Protocol for Supplementary Comparison SIM.T-S7 between INDECOPI, IBMETRO, INTN, INM, INEN, INDOCAL and CENAM for Industrial type Platinum Resistance Thermometers from -60 °C to +400 °C

The instructions and procedures given here shall be followed by the participants in this comparison. By accepting this protocol, the participant laboratories agree to follow the general and technical instructions written in this document and the content in the MRA document *Measurement comparisons in the CIPM MRA* [1].

This SIM Supplementary Comparison is aimed to compare calibrations results of industrial type platinum resistance thermometer (IPRT) in the -60 °C to 400 °C range. In case that a participant laboratory is not able to cover the proposed comparison range, it will notify to the pilot laboratory the range in which it will participate; this notification should be done before the comparison starts.

The method for calibration of IPRTs will be by comparison against the laboratory reference standard in isothermal media.

Participant laboratories will perform their measurements employing their methods and procedures that they currently use for calibration services and the temperature values will be referred to the International Temperature Scale of 1990 (ITS-90).

Seven National Metrology Institutes will participate in this comparison CENAM, México; INDECOPI, Perú; IBMETRO, Bolivia; INTN, Paraguay; INEN, Ecuador; INM, Colombia and INDOCAL, República Dominicana. Where CENAM is the pilot laboratory and INDECOPI acts as coordinating laboratory.

The circulation scheme is in one loop as follows: CENAM – INDECOPI – IBMETRO – INTN – INEN – INM – INDOCAL – INDECOPI – CENAM.

#### **Contacts:**

#### **Pilot Laboratory**:

Centro Nacional de Metrologia, **CENAM** of Mexico Contact: Edgar Mendez Lango, e-mail: <u>emendez@cenam.mx</u>

#### Coordinating Laboratory:

Instituto Nacional de Defensa de la Competencia y de la Protección de la Propiedad Intelectual Servicio Nacional de Metrologia-**INDECOPI** of Perú. Contact: Edwin Guillen, e-mail: eguillen@indecopi.gob.pe

#### **Participants**

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• Instituto Nacional de Tecnologia, Normalización y Metrologia – **INTN** of Paraguay. Contact: Hilda Villa, e-mail: hvilla@intn.gov.py

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## TRANSFER STANDARDS AND TRANSPORT METHOD

CENAM will provide two IPRTs as transfer standards. The transfer standards are two 100  $\Omega$  (nominal value at 0 °C) with four terminals, as described in the following table.

Manufacturer	Sensing Device, Ltd.	Sensing Device, Ltd.		
Model	T100-450-1D	T100-450-1D		
Serial number:	37	43		
Length / cm	35	35		
Diameter / mm	6	6		

Each participant laboratory will have three weeks after reception of the transfer standards to perform its measurements and it will send its results to the pilot laboratory in a period of time not exceeding three weeks after the completion of measurements.

Each participating laboratory will be in charge to transport the transfer standards to the following participant laboratory, if possible by hand but professional carrier may be hired.

### MEASURAND

The measurand for this comparison will be defined through " $W(t_{90})$ " values, where

$$W(t_{90}) = \frac{R(t_{90})}{R_0} \qquad , \qquad (1)$$

where:  $R(t_{90})$ : is the value of the IPRT resistance at temperature  $t_{90}$  and  $R_0$ : is the resistance value of the IPRT at the triple point of water.

For the comparison purposes,  $\Delta t_{90 \text{ lab}}$  will be calculated as follows:

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$$\Delta t_{90_{lab}} = \frac{W_{lab}(t_{90}) - W_{CV}(t_{90})}{\frac{dW(t_{90})}{dt_{90}}}$$
(2)

where,  $W_{\text{lab}}(t_{90})$  is the W value obtained by the laboratory (eq. 1);  $W_{\text{CV}}(t_{90})$  is the comparison value stated by pilot laboratory and  $dW(t_{90})/dt_{90}$  is the first derivative of the W function for the IPRT as was obtained by the pilot in the characterization of the IPRT

#### **MEASUREMENT PROCEDURE**

#### **Preliminary check**

When transfer standards were received at the participant laboratory, they will be inspected to check if they seem to have external and visible damage due to transportation if they seem to be with no damage, the participant laboratory will proceed to measure their  $R_0$  values; participant laboratory will send the corresponding report to the pilot and the coordinating laboratories (Annex 1).

#### Measurements

The electrical resistance measurement will be taken by using four wires electrical connections and the self-heating effect will be measured to report W values at 0 mA condition. Participants laboratories will inform about their standards and equipment used (Annex 2).

#### **Preparation of the IPRTs**

First the IPRT will be placed in a furnace at 400 °C for 10 min, then a new measure of  $R_0$  (0.01 °C) will be taken. The new  $R_0$  value will be compared with the previous one until the scatter of the last three readings be well within 0.001 °C. The participant laboratory will keep record of these measurement results and will be sent to the pilot laboratory upon request.

#### **Comparison points**

Each participant laboratory shall follow its own calibration procedure for calibration of IPRT by comparison. The measurements will be performed from higher to lower temperature values. The calibration points are: 400 °C, 300 °C, 200 °C, 100 °C, 50 °C, -30 °C and -60 °C; the  $R_0$  will be measured after each calibration point (Annex 3).

#### UNCERTAINTY

The uncertainty analysis will be reported in a table as shown in Annex 4. Considering at least the following input values:

The drift of the reference thermometer Stability of  $R_0$  of the reference thermometer. Uncertainty of reference temperature. Uncertainty of the electrical resistance readout. Uncertainty due to temperature gradient in isothermal media. Uncertainty due to repeatability of readings.

#### **REPORT OF RESULTS**

Each participant laboratory will send by e-mail its results to the pilot laboratory in a period of time not exceeding to 3 weeks after completion of calibration measurements. Report of results will include the following information (Annex 2):

- Values of temperature  $t_{90}$ ;  $W_{lab}(t_{90})$  at 0 mA and estimation of uncertainty
- Immersion depth
- Used standards and equipment

Each participant laboratory shall fill out the Annex 3 spreadsheet and it, with its calibration results, will send to the pilot laboratory.

#### **REFERENCE VALUE**

The reference value for the comparison at each calibration point, denoted as  $W_{CV}(t_{90})$ , is the average of the values measured by the pilot laboratory at the beginning  $W_{Beg}(t_{90})$  and at the end of the comparison  $W_{End}(t_{90})$ :

$$W_{CV}(t_{90}) = \frac{W_{Beg}(t_{90}) + W_{End}(t_{90})}{2}$$
(3)

#### **COMPARISON RESULTS**

The pilot laboratory will collect the results of the participant laboratories. The two outcome results to be reported are:

1. **Bilateral differences**, with associated uncertainties at each measured temperature between each participating laboratory and the reference value  $W_{ref}(t_{90})$  using equation (3):

For comparison purposes, bilateral differences between laboratories 1 and 2, denoted by  $\Delta t_{90(1,2)}$ , will be calculated as follows:

$$\Delta t_{90(1,2)} = \frac{W_{lab2}(t_{90}) - W_{lab1}(t_{90})}{\frac{dW}{dt_{90}}}$$
(4)

where:

 $\begin{array}{ll} W_{lab1}(t_{90}) & \text{is the } W \text{ value reported by the laboratory 1 for temperature } t_{90} \text{ using equation (1)} \\ W_{lab2}(t_{90}) & \text{is the } W \text{ value reported by the laboratory 2 for temperature } t_{90} \text{ using equation (1)} \\ dW/dt_{90} & \text{is the first derivative of the } W \text{ function for the IPRT as was obtained by the pilot in the characterization of the IPRT} \end{array}$ 

2. **Standard error** will be calculated in each calibration point for each participant laboratory, using equations (2) and (3):

$$E_{lab,t_{90}} = \frac{\left|\Delta t_{90lab}\right|}{\sqrt{U^2_{lab,t90} + U^2_{CV,t90}}}$$
(5)

where:

- $\Delta t_{90lab}$ : It's obtained from equation (2)
- $U_{\text{lab},t90}$ : It's the expanded uncertainty at temperature  $t_{90}$ , for each participant laboratory for a confidence level of approximately 95%
- $U_{CV,t90}$ : It is the expanded uncertainty at temperature  $t_{90}$ , of the reference value for a confidence level of approximately 95%.

**3. Youden diagrams.** As an auxiliary data Youden diagrams will be generated and inspected. These plots will be prepared for each participant laboratory.

#### **COMPARISON REPORT**

The pilot laboratory will write the Draft A. This Draft will be sent to the participant laboratories, whose will have two weeks to send their observations. If, a participant laboratory does not send observations within this period of time, it will be assumed its agreement with Draft A.

Draft A will be corrected accordingly up to consensus be reached.

The pilot Laboratory will prepare Draft B and it will be submitted to the CCT Key Comparisons Working Group to follow its approval for publication at the BIPM web site.

#### References

1. MRA Document (March 2014): *Measurement comparisons in the CIPM MRA*. CIPM MRA-D05, Version 1.5 <u>http://www.bipm.org/utils/common/CIPM\_MRA/CIPM\_MRA-D-05.pdf</u> Consulted on 2015-03-18.

#### Annex 1 **Format of Reception**

In order to have information about the Comparison development and, if it is necessary to take adequate corrective actions, the participant laboratory must send by e-mail to the pilot laboratory this format after the first measurement at 0.01°C and send to the other participants the reception dates of the thermometers both in its country and in its laboratory.

Thanks for your collaboration.

The travelers standards arrived to my country the day: \_\_\_\_\_ The travelers standards arrived to my laboratory the day:

Were observed damage signals during transport? Yes/Not

If there are damages:

- Are they serious damages? Yes/Not \_\_\_\_\_

- Is it probable that thermometers will be useful? : Yes/Not

- Measurement at 0,01 °C:

  - Resistance: \_\_\_\_\_Ω
    Expanded Uncertainty: \_\_\_\_\_Ω

#### **Comments:**

#### **Participants**

Deliver:

e- mail:

Receiver:

e- mail:

# Annex 2

# **Equipment Used**

## Laboratory name:

Equipment	Description	Manufacturer/ Type	Traceability	Observations
Standard thermometers				
Readout equipment				
Isothermal media				

# Annex 3

# RESULTS

# Laboratory

name:

Temperature $t_{90}(^{\circ}\mathrm{C})$	$W_{ m lab}(t_{90})$ (dimensionless)	Uncertainty of $W_{ m lab}(t_{90})$ (dimensionless)	Immersion depth (cm)

Current intensity: Self-heating at 0,01 °C:

mA
mК

#### Annex 4

# **Estimation of Uncertainty**

# Laboratory name:

#### **Thermometer:**

# **Calibration point:**

Quantity	Quantity estimate	Unit	Probability distribution	Standard uncertainty	Sensibility coefficient	Unit	Contribution to combined uncertainty
Xi	x <sub>i</sub>			$u(x_i)$	$c_i$		$\mathbf{c_i} \cdot \boldsymbol{u}(\boldsymbol{x}_i)$
Laboratory m	easurement sy	stem					
$X_{I}$							
$X_2$							
$X_3$							
$X_4$							
$X_5$							
Readings of the thermometer under calibration							
X <sub>6</sub>							
Characterics of the thermometer under calibration							
<i>X</i> <sub>7</sub>							
Others							

Combined uncertainty: u =Expanded uncertainty (k= \_\_\_): U=