TECHNICAL PROTOCOL

GULFMET.T-S3

Supplementary Comparison on a Platinum Resistance Thermometer Calibration from -70 ºC to 250 ºC

Pilot Lab:

EMIRATES METROLOGY INSTITUTE

U.A.E. National Metrology Institute

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1. Introduction

It is planned to organise a comparison on the calibration of Platinum Resistance Thermometers between the institutes of GULFMET. The comparison will be performed by measuring the electrical resistance of two Platinum Resistance Thermometer for a set of reference temperatures: -70, -35, 0, 50, 100, 200, 250 °C.

Emirates Metrology Institute (EMI) in United Arab Emirates, will be the pilot institute of this comparison. The travelling standards will be provided by the pilot laboratory. EMI will be responsible for monitoring the performance of the standards during the circulation and the evaluation and reporting of the comparison results.

The purpose of comparison is to demonstrate the degree of equivalence between institutes and support CMCs.

The comparison will be carried out in accordance with the “Measurement Comparisons in the CIPM MRA” and “EURAMET Guide on Comparisons” [1, 2].

2. Travelling Standards

The traveling standards are shown in Table 1.

Table 1. Travelling standards

Thermometer 1	Thermometer 2
Manufacturer: Hart Scientific Model: 5615-9 Serial No: 906541 Range: -200 to 420 °C R(0.0 °C) = 100 Ω Suggested measuring current = 1 mA Leads: 4 wires Sheath length: 229 mm Sheath diameter: 4.75 mm Sheath material: Inconel™ 600 Sensor length: 28 mm Sensor location: 6.9 mm ± 3.3 mm from tip	Manufacturer: Hart Scientific Model: 5615-12 Serial No: 883912 Range: -200 to 420 °C R(0.0 °C) = 100 Ω Suggested measuring current = 1 mA Leads: 4 wires Sheath length: 305 mm Sheath diameter: 6.35 mm Sheath material: Inconel™ 600 Sensor length: 28 mm Sensor location: 6.9 mm ± 3.3 mm from tip

3. Participating Institutes

The pilot and coordinator institute for this comparison is Emirates Metrology Institute (UAE). The participating institutes and contact persons with their addresses are given in Table 2.

Table 2. Participating Institutes

Country	Institute	Acronym	Shipping Address	Contact Person
United Arab Emirates	Emirates Metrology Institute (pilot lab)	EMI	Block H, CERT Technology Park, 881, Sultan Bin Zayed The First Street, PO Box 853, Abu Dhabi, United Arab Emirates	Dr. Miltiadis Anagnostou miltiadis.anagnostou@qcc.abudhabi.ae Tel :+97124035981
Turkey	TÜBİTAK Ulusal Metroloji Enstitüsü	TÜBİTAK UME	TÜBİTAK Ulusal Metroloji Enstitüsü (UME) TÜBİTAK Gebze Yerleşkesi Barış Mah. Dr. Zeki Acar Cad. No:1 41470 Gebze-Kocaeli, TURKEY	Dr. Murat Kalemci murat.kalemci@tubitak.gov.tr
Saudi Arabia	SASO The National Measurement and Calibration Center	SASO NMCC	Saudi Standards, Metrology and Quality Organization of The Kingdom of Saudi Arabia (SASO) Riyadh 11471, P.O. Box 3437 KINGDOM of SAUDI ARABIA	Eng. Oqab N. Alotaibi Tel: +966 11 2529737 and Eng. Rakan O. AlNefaie r.nefaie@saso.gov.sa Tel: +966 11 2529767
Qatar	Qatar Standards	QGOSM	Qatar General Organization for standardization Industrial area Rd., Abu Hamour, P.O. Box 23277 Doha	Mrs. Aisha Al Suwaidi amsuwaidi@mme.gov.qa
Oman	Metrology & Assay of Precious Metals Ministry of Commerce and Industry	MOIC		Mrs. Faiza Hamed Faiza3066@gmail.com

By their declared intention to participate in this comparison, the laboratories accept the general instructions and the technical protocol written down in this document and commit themselves to follow strictly the procedures of the protocol.

Once the protocol and the list of participants have been approved, no change can be made without prior agreement of all participants.

4. Time Schedule

The time schedule for the comparison is given in Table 3 below:

Table 3. Time Schedule

Acronym of Institute	Country	Starting Date	Approximate time for measurements and transportation
EMI	United Arab Emirates	01.09.2019	40 days
SASO NMCC	Saudi Arabia	13.10.2019	40 days
TÜBİTAK UME	Turkey	25.11.2019	40 days
QGOSM	Qatar	05.01.2020	40 days
Ministry of Commerce and Industry	Oman	16.02.2020	40 days
EMI	United Arab Emirates	27.03.2020	40 days

Any deviation in the agreed plan should be approved by the pilot institute.

If for some reason, the measurement facility is not ready or customs clearance takes too much time in a country, the participating laboratory must contact the Pilot laboratory immediately.

5. Transportation of Travelling Standard

The travelling standards are packed in a carton box of size (50 x 20 x 20) cm and total weight of 3 kg. The transport box can be easily opened for customs inspection.

Each participant is responsible for the transportation of the travelling standards to the next participant.

Any participant has to notify the pilot lab upon receiving the thermometers and report their resistance at the ice / water triple point and their general condition.

Stability of the instruments during the comparison will be monitored from the resistance measurements of the participating labs at the ice point / triple point of water.

Each institute will have about 40 days available for their measurements and for the transportation of the standard to the next participant.

5.1. Failure of Travelling Standard

In case of any damage or malfunction of the travelling standards, the Pilot institute (EMI, UAE) should be informed as soon as possible.

5.2. Financial aspects

Each laboratory is responsible for any damage or loss of the artefacts from the point of receipt at their site until the artefacts are signed for on receipt at the next laboratory. The insurance

value of the artefacts is 2000 EUR. Each participant institute is responsible for the cost of shipping to the next participant including any customs charges.

6. Measurement Quantities and Points

Main quantity which must be measured is the electrical resistance of the PRTs at the reference temperatures of the comparison.

The measurement temperature points of the comparison are:

0.01, -70, -35, 0.01, 50, 100, 200, 250, 100, 0.01 °C

The electrical resistance measurements will be performed with four terminals using 1mA current. If another current is used, it must be specified and the self-heating has to be determined.

Participants may perform any initial checks on the platinum resistance thermometers that would be performed for a normal calibration. In the case of an unexpected instrument characteristics, the pilot shall be informed as soon as possible.

An initial ice / water triple point resistance measurement has to be performed and communicated to the pilot lab. The pilot lab will decide whether an annealing of the thermometer is necessary or not. If an annealing is decided, then an annealing for 4 hours at 260 °C has to follow and the ice / water triple point of the thermometers has to be measured again. Equivalent change of the thermometers should be less than 10 mK. If change is larger than this number, then the annealing has to be repeated.

Every lab has to calibrate the thermometers in comparison with its own reference thermometers using baths or block calibrators. The measurements have to be performed from the lower to the higher temperatures and then down to 100 °C and 0.01 °C to account for the hysteresis of the thermometers. At each set-point, and after the stability of the temperature has been reached, at least 10 measurements have to be taken in a period of 10 to 20 minutes for the reference temperature and the electrical resistance of the test thermometers. The mean and standard deviation of these measurements have to be determined and reported.

If the scope/capability of a laboratory does not cover the whole range of this comparison, the laboratory is allowed to limit measurement values according to their capability. In this case, the Pilot institute (EMI, UAE) should be informed by the participant laboratory.

7. Measurement Uncertainty

The uncertainty of measurement must be calculated according to the JCGM 100 “Guide to the Expression of Uncertainty in Measurement” [3] for the coverage probability of approximately 95%.

All contributions to the measurement uncertainty should be listed in the report submitted by each participant.

Even though the contributions to the uncertainty are specific to the measurement method used, it may be useful to consider the list of uncertainty sources given below.

1. The Type A standard uncertainty of reference temperature and PRT resistance output
2. The estimated uncertainty of the reference temperature including calibration and drift of reference thermometer also calibration, drift and resolution of the reading instrument used to read reference thermometer.
3. The estimated uncertainty term from the bath instability
4. The estimated uncertainty term from the bath inhomogeneity
5. The estimated uncertainty relating to the short-term stability of the travelling standard at the time of measurement
6. The estimated uncertainty relating to the hysteresis of the travelling standard (Difference between measurements in ascending and descending order).

This is not a complete list and should be extended with uncertainty contributions that are specific for the participant's measurement system. The labs can get advice for the uncertainty terms required from the guideline DKD-R 5-1 [4].

Uncertainty terms related to electrical resistance must be converted to equivalent temperature terms using the corresponding sensitivity of the reference or test PRT.

Each participant is required to submit detailed analyses of uncertainty for their standards. A list of all significant components of the uncertainty budget should be evaluated, and should support the quoted uncertainties. A template for reporting uncertainty of measurement is given in Appendix 1.

8. Reporting of Results

The results should be sent to the pilot institute at the latest six (6) weeks after completing the measurements.

The templates for reporting results are provided in Appendix 1:

- In Table A. "MEASUREMENT RESULTS", participants should report the actual mean of their reference temperatures and the standard deviation of these measurements, also they should report the actual mean of the PRT resistance values at these temperatures and their standard deviation.

- In Table B. "RESULTS REDUCED TO NOMINAL TEMPERATURES", participants should calculate, using their measurements, and report the PRT resistance values at the exact nominal points of the comparison. This will help us compare the results of the labs, since during calibration every lab measures at a slightly different temperature, close to the nominal value. In the same page, an average sensitivity dR/dt of the resistance vs temperature curve is given, calculated from the respective curves of both thermometers. These values can be used by the participants to calculate the PRT resistance values at the exact nominal points of the comparison. Also the participants

have to calculate the combined standard uncertainty of their measurements and report in this table (last column).

- In Table C. “UNCERTAINTY CALCULATION”, participants should present an analytical evaluation of their uncertainty. If the uncertainty is not the same in all measurement points, they should provide more than one tables covering the whole range of measurements.
- In Table D. “EQUIPMENT INFORMATION”, participants should provide information for their equipment used and its traceability.

Exclusion of a participant's results from the report may occur if the results are not available in time to prepare the draft report.

Participants should avoid sharing any data until the Draft report is completed by the pilot.

9. Calculation of the Comparison Reference Values

Actually the resistance ratio $W_{lab}(t) = \frac{R(t)}{R(0.01\ ^oC)}$ of each thermometer, for every lab, will be compared with the weighted mean value calculated from all labs:

$$W_{wmean}(t) = \frac{\left[\frac{1}{u(W_{lab1}(t))^2}\right] * W_{lab1}(t) + \left[\frac{1}{u(W_{lab2}(t))^2}\right] * W_{lab2}(t) + \cdots}{\left[\frac{1}{u(W_{lab1}(t))^2}\right] + \left[\frac{1}{u(W_{lab2}(t))^2}\right] + \cdots} \quad (1)$$

As *R(t)* value for every lab will be taken the average of the resistance value of the ascending measurements reported by the lab for every nominal temperature set point (Table B in the Appendix 1.). As *R(0.01 ^oC)* value for every lab, will be taken the average of the three values reported by the lab in Table B, Appendix 1. The standard uncertainty of W_{lab} will be calculated combining the standard uncertainties of *R(t)* and *R(0.01 ^oC)* reported by the labs.

The deviation of $W_{lab}(t)$ values from the $W_{wmean}(t)$ will be calculated using equation 2:

$$\Delta W_{lab}(t) = W_{lab}(t) - W_{wmean}(t) \quad (2)$$

In case that some lab has highly deviating results, which can significantly alter the weighted mean of the comparison, its results will not be used in the determination of the weighted mean.

The pilot will make an assessment of any drift in the travelling standards during the comparison. The assessment will be based on initial and final measurements done by the pilot. If drift is found, this will be taken into account in the uncertainty of the reference value of the comparison u_{drift}.

The normalized deviation for every lab will be calculated using the following formula:

$$E_{n-lab} = \frac{\Delta W_{lab}(t)}{2 * \sqrt{u(W_{lab}(t))^2 - u\left(W_{wmean}(t)\right)^2 + u_{drift}^2}}$$

(3)

Note that this comparison is an RMO supplementary comparison so the W_{wmean} values generated are used only as a baseline for reporting the results, and they have no particular meaning of reference values in the sense of the key comparisons.

10. Final Report of the Comparison

The pilot institute is responsible for the preparation of the comparison report.

The draft version of the comparison report will be issued within 12 weeks after receiving the last participant report by the pilot institute.

In preparing Draft A, the pilot lab may ask participants to check any apparently anomalous reported results, without revealing their magnitudes. The pilot will also check the uncertainty budgets reported and make sure that they are correctly derived.

Draft A report will be sent to each participant for discussion and approval. This draft A will be confidential to the participants. The participants will have one week to send their comments. After one week, Draft A Report will become the draft B report and finally the Final Report.

The Final Report will form the basis for the publication of the results in the Key Comparison Data Base of BIPM.

11. References

[1] Measurement Comparisons in the CIPM MRA, MRA-D-05, V1.6

[2] EURAMET Guide on Comparisons, EURAMET Guide No. 4, V1.0 (05/2016)

[3] JCGM 100, “Guide to the Expression of Uncertainty in Measurement” (GUM), First edition, September 2008 (available on the BIPM website: http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf).

[4] Guideline DKD-R 5-1, “Calibration of Resistance Thermometers”, Edition 10/2003, English translation 02/2009. The guideline was withdrawn on December 31, 2009 and replaced by: Richtlinie DAkkS- DKD-R 5-1, Kalibrierung von Widerstandsthermometern, Neuauflage 2010, in German language.

APPENDIX 1. TEMPLATES for SUBMISSION of RESULTS

A. MEASUREMENT RESULTS

Name of the Laboratory: **Date of Measurements:** **Thermometer:**

Reference temperature (°C)	Standard deviation of ref. temp. measurements (°C)	PRT Resistance (ohm)	Standard deviation of PRT resistance measurements (ohm)	Number of Measurements

Current: **……** mA Self-heating at 0 °C: **……** mK

Immersion during measurements: …….. cm

B. RESULTS REDUCED TO NOMINAL TEMPERATURE

Name of the Laboratory: **Thermometer:**

Nominal temperature (°C)	PRT Resistance at the nominal temperature (ohm)	Standard uncertainty (°C)
0.01		
-70		
-35		
0.01		
50		
100		
200		
250		
100		
0.01		

Resistance results must be reduced to the exact (nominal) temperatures points of the comparison (first column).

The table below is giving an average slope of the R vs t curve for the two thermometers at the different set points of the comparison. The slope can be used to reduce the resistance results of the participants to the exact (nominal) temperature points of the comparison.

Nominal temperature (°C)	dR/dt (ohm / °C)
-70	0.4080
-35	0.4030
0.01	0.3987
50	0.3926
100	0.3866
200	0.3748
250	0.3689

C. UNCERTAINTY CALCULATION

Name of the Laboratory: *Points of calibration:*

Quantity X_i	Estimation of the quantity x_i	Unit	Probability Distribution	Standard Uncertainty	Sensitivity coefficient c_i	Unit	Contribution to the combined uncertainty $u(x_i)$ [^oC]

Combined uncertainty: u=
Expanded uncertainty for k=2: U=

D. EQUIPMENT INFORMATION

Name of the Laboratory:

Equipment	Description	Manufacturer	Type	Traceability
Standard Thermometers				
Measuring Instruments				
Isothermal Media				