Comparison of the calibration of noble metal thermocouple from 0 ºC up to 1200 ºC

SADCMET ILC/TEM – 002/2019

Prepared by

Dr Efrem K Ejigu / B Chibaya
Comparison of the calibration of noble metal thermocouple from 0 ºC up to 1200 ºC

SADCMET ILC/TEM – 001/2019
1. INTRODUCTION

SADCMET temperature technical committee has decided to organise an intercomparison in different temperature parameters. One of the parameters is thermocouple where members show a great interest in participating.

The objective of this comparison is to establish a degree of equivalence between the participants for the calibration of noble metal thermocouple by comparison from 0 °C up to 1200 °C.

Scientific and Industrial Research and Development Centre’s National Metrology Institute (SIRDC-NMI) has shown interest to pilot this comparison and was agreed by the members at the workshop held in Botswana in February 2019.

The Comparison project is funded by PTB

2. PARTICIPANTS

The responsibility of the pilot laboratory which is SIRDC-NMI is to collect the measurement information from all participants and perform the analysis of the comparison data and elaborate the reports four months after the last participant report was received.

The National Metrology Institute of South Africa (NMISA) will assist to

- Organize to buy a stable noble metal thermocouple (Type R or S) with the following characteristics from PTB fund to perform the measurements:
  i. length of the alumina sheath: 700 mm
  ii. length of the thermoelements: 2000 mm
  iii. maximum outer diameter: 7 mm

- The National Metrology Institute of South Africa (NMISA) will assist to prepare the protocol, perform the initial and final measurement of the thermocouple to provide a reference value and give advice when the pilot prepares the report

Each participant laboratory should:

- perform the measurements according to the rules of this protocol and their own calibration procedure,
- Air or hand-carry the thermocouple to the next participant in due time,
- send to the pilot laboratory a report using the forms included in the annex one month after the completion of its measurements.

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Table 1. shows the contact details of the comparison participants.

<table>
<thead>
<tr>
<th>INSTITUTE</th>
<th>COUNTRY</th>
<th>Contact Person</th>
<th>e-mail/Phone</th>
<th>Address</th>
</tr>
</thead>
<tbody>
<tr>
<td>NMISA</td>
<td>SOUTH AFRICA</td>
<td>Dr. Efrem K. Ejigu</td>
<td><a href="mailto:eejigu@nmisa.org">eejigu@nmisa.org</a> <a href="mailto:rmnguni@nmisa.org">rmnguni@nmisa.org</a></td>
<td>Meiring Naudé Road Brummeria Pretoria South Africa</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ms Regina Mnguni</td>
<td></td>
<td></td>
</tr>
<tr>
<td>OCC</td>
<td>DRC</td>
<td>Ms Mpiana Kaja Nady</td>
<td><a href="mailto:nadyocpe@gmail.com">nadyocpe@gmail.com</a></td>
<td></td>
</tr>
<tr>
<td>Eswatini Standards Authority)</td>
<td>Kingdom of Eswatini</td>
<td>Ms Cebsile Bhembe</td>
<td><a href="mailto:cbhembe@swasa.co.sz">cbhembe@swasa.co.sz</a></td>
<td></td>
</tr>
<tr>
<td>NMI-SIRDC</td>
<td>Zimbabwe</td>
<td>Blessing Chibaya</td>
<td><a href="mailto:bchibaya@sirdc.ac.zw">bchibaya@sirdc.ac.zw</a> <a href="mailto:bchibaya@gmail.com">bchibaya@gmail.com</a></td>
<td>1574 Alpes Road, Technology Drive Hatcliffe P.O. Box 6640 Harare</td>
</tr>
<tr>
<td>ZMA</td>
<td>Zambia</td>
<td>Ms Careen Mutembo</td>
<td><a href="mailto:karenmutembo@gmail.com">karenmutembo@gmail.com</a></td>
<td>Lechwe House, Freedom Way P.O. Box 50259 Lusaka</td>
</tr>
<tr>
<td>DSQA</td>
<td>Lesotho</td>
<td>Ms Qenehelo Lenka</td>
<td><a href="mailto:Qenehelo2008@yahoo.com">Qenehelo2008@yahoo.com</a></td>
<td>Ministry of Trade and industry, LNDC Building, Kingways Road, Lesotho</td>
</tr>
<tr>
<td>TBS</td>
<td>Tanzania</td>
<td>Adam Ziagi</td>
<td><a href="mailto:adam.ziagi@tbs.go.tz">adam.ziagi@tbs.go.tz</a></td>
<td></td>
</tr>
<tr>
<td>BOBS</td>
<td>Botswana</td>
<td>Tebogo Kajane</td>
<td><a href="mailto:kajane@bobstandards.bw">kajane@bobstandards.bw</a> <a href="mailto:nkgare@bobstandards.bw">nkgare@bobstandards.bw</a> <a href="mailto:tshaila@bobstandards.bw">tshaila@bobstandards.bw</a></td>
<td>Private Bag B0 48 Gaborone</td>
</tr>
<tr>
<td>Qualidade-INNOQ</td>
<td>Mozambique</td>
<td>Mr Elves Emilio Djedje</td>
<td><a href="mailto:elvesdjedje@gmail.com">elvesdjedje@gmail.com</a></td>
<td>Av. Moçambique, Parcela 7168/D1/7[,] Bairro do Zimpeto[,] C.P: 2983; Maputo - Moçambique</td>
</tr>
<tr>
<td>MSB</td>
<td>Mauritius</td>
<td>Mr Christian Ng Ha Kwong</td>
<td><a href="mailto:jchrisng@gmail.com">jchrisng@gmail.com</a></td>
<td>Mauritius Standards Bureau, Villa Road, Moka, Republic of Mauritius</td>
</tr>
<tr>
<td>NSI</td>
<td>Namibia</td>
<td>Mr Simasiku Matali</td>
<td><a href="mailto:Matalis@nsi.com.na">Matalis@nsi.com.na</a></td>
<td></td>
</tr>
<tr>
<td>MBS</td>
<td>Malawi</td>
<td>Mr Crighton Marorongwe</td>
<td>crightonmarorongwe@mb smw.org</td>
<td></td>
</tr>
</tbody>
</table>

*Comparison of the calibration of noble metal thermocouple from 0 °C up to 1200 °C*

**SADCMET ILC/TEM – 001/2019**
3. ARTEFACT

The following artefact is purchased and will be used in the comparisons

<table>
<thead>
<tr>
<th>Thermometer</th>
<th>S/N</th>
<th>Model</th>
<th>Manufactur er</th>
<th>TYPE</th>
<th>Diameter (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>THERMOCOUPLE</td>
<td></td>
<td></td>
<td></td>
<td>R</td>
<td></td>
</tr>
</tbody>
</table>

4. COMPARISON SCHEDULE

The proposed start date is May 2019. Table 2 presents the schedule. Each participant counts on 2 months to perform the measurements.

<table>
<thead>
<tr>
<th>Institute</th>
<th>Planned date</th>
<th>Actual date</th>
</tr>
</thead>
<tbody>
<tr>
<td>NMISA South Africa</td>
<td>May 2019</td>
<td>May 2019</td>
</tr>
<tr>
<td>SIRDC-NMI Zimbabwe</td>
<td>June 2019</td>
<td>June 2019</td>
</tr>
<tr>
<td>Botswana</td>
<td>July 2019</td>
<td></td>
</tr>
<tr>
<td>Namibia</td>
<td>August 2019</td>
<td></td>
</tr>
<tr>
<td>Zambia</td>
<td>September 2019</td>
<td></td>
</tr>
<tr>
<td>Tanzania</td>
<td>October 2019</td>
<td></td>
</tr>
<tr>
<td>Malawi</td>
<td>November 2019</td>
<td></td>
</tr>
<tr>
<td>Mozambique</td>
<td>December 2019</td>
<td></td>
</tr>
<tr>
<td>Mauritius</td>
<td>January 2020</td>
<td></td>
</tr>
<tr>
<td>Seychelles</td>
<td>February 2020</td>
<td></td>
</tr>
<tr>
<td>South Africa</td>
<td>March 2020</td>
<td></td>
</tr>
<tr>
<td>Zimbabwe</td>
<td>April 2020</td>
<td></td>
</tr>
</tbody>
</table>

5. PROCEDURE

5.1. Initial inspection

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As soon as the equipment is delivered and the thermocouple unpacked and inspected, the participant should inform the coordinator by e-mail. If the equipment has any visible damage due to transportation, this must be reported to the pilot before the calibration begins using the form attached in the appendix.

5.2. Thermocouple homogeneity tests

Before starting the calibration, each participant should determine the thermocouple homogeneity:

- Immersion test in an oil bath at 150 - 200 °C. Particular care should be taken to avoid contamination of the wires. The thermocouple will be introduced in an appropriate protective alumina sheath in order to avoid direct contact with the liquid and cleaned with alcohol and distilled water after the test.

5.3. Measurements

The participating laboratory should not dismantle the thermocouple and the thermocouple should not be subject to any further annealing before or after the calibration. Additionally, participants should not perform any heat treatment to the thermocouple. To maintain its thermoelectric stability, the exposure at high temperatures must be as short as possible.

Conduct measurement at the following temperatures by using comparison method: 200 °C, 400 °C, 600 °C, 800 °C, 1000 °C and 1200 °C

6. Reference Value

The comparison reference value will be the average of the initial and final measurements by NMISA.

Each participants measurement performance can be assessed based on the En values. En values can help to identify discrepancies amongst participant’s measurement data which is aimed at fulfilling one of MRA requirements. Usually En values that are above 1 indicate the discrepancy. The normalized error En can be calculated by the following equation.

\[ E_n = \frac{\Delta T_{NMI-CRV}}{u_{NMI-CRV}(k = 2)} \]

\[ \Delta T_{NMI-CRV} = \Delta T_{NMI} - \Delta T_{CRV} \]

\[ u_{NMI-CRV}(k = 2) = \sqrt{(u(\Delta T_{NMI}))^2 + (u(\Delta T_{CRV}))^2} \]

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\[ \Delta T_{CRV} = \Delta T_{\text{REFERENCE NMI}} \]

\[ u(\Delta T_{CRV}) = u\left(\Delta T_{\text{REFERENCE NMI}}\right) \]

7. UNCERTAINTIES

The laboratory should perform the uncertainties estimation according to its usual procedures, however at least the following sources of uncertainty will be taken into account:

- \( t_{CP} \): uncertainty due to the temperature determination of the calibration point.
- \( E_{vc} \): uncertainty due to the voltmeter calibration.
- \( E_{vr} \): uncertainty due to the voltmeter resolution.
- \( E_{vd} \): uncertainty due to the voltmeter drift.
- \( t_{0C} \): uncertainty due to the thermocouple cold junction.
- \( E_{\text{Hom}} \): uncertainty due to the thermocouple homogeneity.

Any other source of uncertainty, like the influence of the isothermal enclosure and/or the heat flux along the thermocouple wires, should be added to the uncertainty budget with a brief explanation of their admission.

8. REPORTING OF DATA

The participating laboratories should send the following measurement results and information to the coordinator:

- details of instrumentation
- temperature profiles of furnaces used at the highest calibration point by comparison
- results of the thermocouple homogeneity tests
- measured emfs at the calibration points
- uncertainty budget of the measurements

The information should be reported using the excel sheet formats in the format given in Appendices A & B.

9. REFERENCES

http://www.bipm.org/utils/common/CIPM_MRA/CIPM_MRA-D-05.pdf

[2] AFRIMETS TS-7 protocol

[3] APMP-T-S3-03 protocol

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Appendix A

ARTEFACT DISPACHED

To: Thermometry Department                                    Fax No.: 

Comparison of Thermocouple  S/No. ______________________  

The Thermocouple was dispatched from ____________________________  

On:……/……/……  

The condition when it was dispatched was  

   ( ) in good physical and working order  

   ( ) damaged (please explain)…………………………….

Participating Laboratory: 

Contact Person: 

Tel: 
Fax:  

In order to monitor the comparison, we kindly ask each participating laboratory, upon dispatching of the artefact, to fill in this confirmation form and return it to:

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Appendix B

ARTIFACT RECEIVED

To: Thermometry Department

Comparison of Thermocouple  S/No. __________________________

The Thermocouple was received at………………………………

On:……/…… /……

The condition when it was received was

  ( ) in good physical and working order

  ( ) damaged (please explain)………………………………

Participating Laboratory:

Contact Person:

Tel:

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Fax:

In order to monitor the comparison, we kindly ask each participating laboratory, upon arrival of the artefact, to fill in this confirmation form and return it to:

Mr. Blessing Chibaya

SIRDC-NMI
Thermometry Department
1574 Alpes Road, Technology Drive Hatcliffe
P.O. Box 6640
Harare

Mobile Phone: +263772107300
Telephone Number: +263242860346
Fax: +263242860350
E-mail: bchibaya@sirdc.ac.zw or bchibaya@gmail.com

Appendix C: Measuring equipment and standards used in the comparison
Laboratory Name: __________________________

<table>
<thead>
<tr>
<th>Device</th>
<th>Type</th>
<th>Manufacturer</th>
<th>Serial number</th>
<th>Description</th>
<th>Immersion (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard used for reference temperature</td>
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<td></td>
<td></td>
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</tr>
<tr>
<td>AC bridge/DVM for SPRT measurements</td>
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<tr>
<td>Ice-Point used</td>
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<td></td>
<td></td>
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</tr>
<tr>
<td>DVM for Test TC</td>
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<td></td>
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</tr>
<tr>
<td>Scanner (if used)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Enclosure used</td>
<td></td>
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</tr>
</tbody>
</table>

* Note: Please write about the traceability of the standard used

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Appendix D: Calibration Data of the Thermocouple

Serial Number ___________  Inhomogeneity ___________________
Name of the Laboratory:_______________________________________

<table>
<thead>
<tr>
<th>Temperature/°C</th>
<th>$E_{\text{ref}}$/ μV</th>
<th>E/ μV</th>
<th>$E - E_{\text{ref}}$/ μV</th>
<th>Uncertainty/°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>200</td>
<td></td>
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<tr>
<td>400</td>
<td></td>
<td></td>
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<td></td>
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<tr>
<td>600</td>
<td></td>
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</tr>
<tr>
<td>800</td>
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<td></td>
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</tr>
<tr>
<td>1000</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1200</td>
<td></td>
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</tr>
</tbody>
</table>

* $E_{\text{ref}}$ is the reference emf given by IEC or ASTM

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